VOL. XX. 1911

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VOLUME XX.

TWENTIETH GENERAL MEETING

TORONTO, CANADA, SEPT. 21, 22, 23, 1911.

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LEHIGH UNIVERSITY, SOUTH BETHLEHEM, PA.
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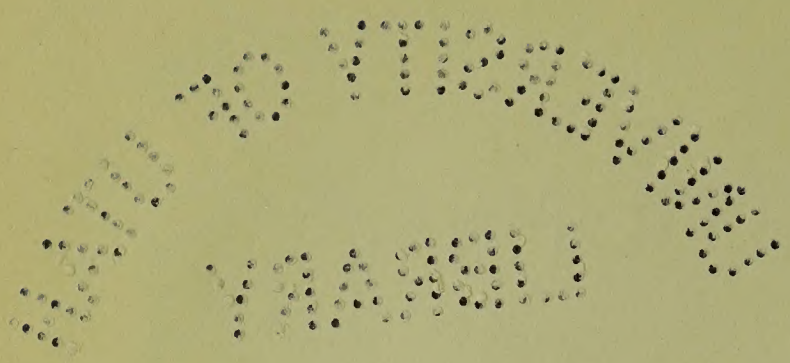

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VOLUME XX. 1911.

TRANSACTIONS

OF THE

AMERICAN ELECTROCHEMICAL SOCIETY

PROCEEDINGS

CONDENSED MINUTES OF THE TWENTIETH GENERAL MEETING OF THE SOCIETY, HELD IN TORONTO, CANADA, SEPTEMBER 21, 22, 23, 1911.

Number of members registered, 82; guests, 38; total, 120.

SESSIONS OF SEPTEMBER 21st.

The morning session was called to order at 10 o'clock, in the Lecture Room 22, Chemistry and Mining Building, School of Practical Science, Toronto University, President W. R. Whitney in the Chair, Mr. Carl Hering, Secretary *pro tem.*

Papers by T. D. Robertson, F. A. J. FitzGerald, C. V. Slocum, A. J. Jones, E. T. Dittus and R. G. Bowman (read in abstract by E. F. Roeber), Albert Hiorth (read in abstract by Carl Hering), were presented and discussed, as printed in full in these Transactions.

The afternoon session was called to order in the same room at 2.30 o'clock, President W. R. Whitney in the Chair.

Papers by T. G. Gibson, Ching Yu Wen and Edward F. Kern (read by W. D. Bancroft), H. K. Richardson and F. D. Taylor (read by F. A. Lidbury), W. C. Arsen (read in abstract by C. A. Hansen), R. Winne and C. Dantsizen (read by C. Dantsizen), E. F. Northrup, C. F. Burgess and J. Aston (read by O. P.

Watts) were presented and discussed, as printed in full in these Transactions.

President Whitney called upon Mr. F. A. J. FitzGerald to present the report of the Special Committee on Improvement of Standard of Papers read before the Society and printed in its Transactions:

NIAGARA FALLS, N. Y., July 5, 1911.

DR. W. R. WHITNEY,
President Am. Electrochemical Society,
Research Laboratory, General Electric Co.,
Schenectady, N. Y.

DEAR SIR:

Your Committee, appointed "to consider and report whether a higher standard of selection of papers read at the meetings of the Society or published in the Society's Transactions, should be observed, and in the event of an affirmative opinion being arrived at to consider and recommend appropriate measures for the attainment and maintenance of a higher standard," answers the question put to it in the affirmative, and transmits herewith its recommendations.

Of the numerous letters which have been received, and which have been of the greatest assistance to your Committee, it is thought desirable to quote from two, which may be regarded as embodying the experience of the American Institute of Electrical Engineers and of the American Chemical Society, respectively:

"A national society such as the American Electrochemical Society has a double function to perform.

"First, is the advancement of the applied science with which it deals through the presentation of papers presenting new discoveries and new methods, and otherwise building up transactions which are representative of the highest and best in its line.

"Second, it should contribute to the general advancement of its department by a general educational process through the presentation of papers dealing with more elementary principles both from their theoretical side and in their application. It should give descriptions of processes in practical operation and should be of interest to beginners and to general practitioners who might be but little interested in the scientific and more advanced papers.

"Both of these functions are important and neither should be allowed to exclude the other. On the one hand it is not desirable to have the printed transactions filled with elementary and descriptive matter, and, on the other hand, the contents should not be limited to advanced papers and discussions in which the majority of those who attend might take but little interest.

"The American Institute of Electrical Engineers encountered the same problem. In a general way the following may be given as its solution:

"Papers of all kinds are received. Its branches, of which there are two dozen among its members and three dozen in technical schools and universities, have papers which are of interest and profit to the varied membership of these many sub-organizations.

"From these papers, those which are of permanent value are printed both in its monthly proceedings and also in its bound annual transactions. Furthermore, papers which may be of general interest, though not ranking in permanent value with the first class, are selected and printed in the monthly proceedings. The papers, therefore, may be considered of three grades:

(a) Those merely of local interest which are not printed at all.

(b) Those of permanent value which are printed both in the monthly proceedings and in the annual transactions.

(c) The intermediate grade which is published in the monthly proceedings only.

"The American Electrochemical Society is doing well to consider whether a higher standard of papers should not be maintained, but, in doing so, it should not overcome the good results which can come from a society of this kind through the general instruction and education of its less technical members by the dissemination of papers which may fall short of a high standard of technical excellence.

"The general subject of electrochemistry is new. It brings together electrical men and chemical men and it is introducing new processes. All this newness makes more important the carrying out of the general educational ideas which I have expressed above.

"You must have some central committee or authority to pass on papers that will cut out papers that are worthless. When this is known and rigidly adhered to you will have little difficulty with poor papers, but in the meantime—God help the committee!"

Your Committee recommends:—

1. That the duties of the Committee on Papers be divided, and the Committee abolished;

That the Secretary be entrusted with the organization of machinery for securing the submission of papers—the systematizing of this machinery, with the establishment of a card-index, are suggested in this relation;

That the duty of passing on all papers submitted, whether for presentation or publication, be allotted to a Publication Committee;

2. That there should be a strong Committee on Publication to whom all papers should be referred before presentation or publication.

Such Committee should have complete discretion as to what should or should not be accepted, and should as far as possible be unhampered by rulings of the Board of Directors; for instance, the present rule as to "previous publication" should be rescinded, and the Committee given a free hand in this and similar matters.

Such Committee should, where thought desirable, assist authors by

suggestions which would improve the form of their papers, without, however, discouraging them by undue criticism.

For obvious reasons no member of such Committee should pass on his own paper.

Such committee should deal with papers as promptly as possible after their submission, and particularly where the author desires early publication of advance copies.

A record should be kept of all papers submitted to such Committee, and of the action taken thereon.

Your Committee desires to state its emphatic opinion that the success of the scheme embodied in these recommendations must depend upon the *personnel* of the Committee on Publication, and upon the support accorded to it, in the discharge of its difficult and important duties, by the Society at large.

3. That, in general, no paper should be presented at a meeting unless it has been submitted in full at or before a date set by the Publication Committee as suitable to allow its consideration by that Committee and the printing of advance copies before the meeting.

In exceptional cases the President might authorize a paper, not previously submitted to the Publication Committee, to be read; such permission, however, should not bind the Society in any way as to printing or publication.

4. That the main interest of each, or at any rate of every alternate meeting, be concentrated on one topic to which at least one session should be devoted.

The success of the Symposium on Iron and Steel shows what can be done in this direction. Obviously the topic must be chosen well in advance and the organization must in each case be in the hands of one peculiarly able to get together the proper papers. A promising suggestion is that the President should organize a symposium each year.

5. That the duties of the Editor of the Transactions be separated from those of the Secretary of the Society. Neither the Editor of the Transactions nor the Secretary of the Society should serve on the Publication Committee.

6. That of the papers accepted by the Publication Committee for presentation at the meetings, only those of permanent scientific value be selected for publication in the Transactions; that those of purely immediate interest, or of educational import, be published in a suitable journal.

This recommendation does not contemplate the publication in that journal of all work of sufficient permanent value to be selected for the Transactions; though such of this work as might also be of immediate interest would be so published in full or in abstract.

In the event of the relations suggested above being established, it would be desirable that the Editor of that journal of the Society should also edit the Transactions of the Society.

7. That advance copies of such papers as are accepted by the Publication Committee be printed as soon as possible for distribution before the meeting, and that the author receive 25 of these copies gratis and have the right to order as many as he may wish at the regular rates; that the copyright of all papers printed in the transactions remain vested in the Society, but that, in general, permission be granted by the President to the author or to other journals to reprint in part or in whole, due acknowledgment of source being given.

8. That three classes of papers be regarded as undesirable:

a. Those containing no information or obviously incorrect information.

b. Those containing only information already easily accessible.

c. Those of an advertising nature not counter-balanced by adequate scientific information and treatment.

It may be desirable, however, to accept authors' abstracts of papers already published elsewhere (with adequate reference) for the purpose of discussion. Papers already published but not easily accessible, should, if otherwise desirable, be generally accepted.

9. That the Publication Committee should edit the "discussions" and eliminate from them all irrelevant and redundant matters as well as matter which is merely a duplication of previous discussions.

Respectfully submitted,

F. A. J. FitzGerald, Chairman.
W. D. Bancroft.
A. H. Hooker.
F. A. Lidbury.
W. Lash Miller.

The following resolutions presented by Mr. E. R. Taylor were unanimously approved:

There are ten thousand chemists in the United States. They are largely concerned in the working out of economic problems and the best utilization of all substances capable of adding to our material prosperity. Many of these chemists are members of the American Chemical Society, The American Electrochemical Society, The American Institute of Chemical Engineers and The Society of Chemical Industry. All of which Societies are deeply interested in the best conservation of our natural resources and are in full sympathy with the objects of this Congress.

Chemical industries are already making large demands for cheap water power, which demand is constantly on the increase. Forty-six percent of the total power utilized at Niagara Falls is absorbed in electrochemical industries.

The United States has the honor of entertaining the Eighth International Congress of Applied Chemistry in 1912, from September 4th to the 13th, inclusive.

Section XI b has to do with Political Economy and Conservation of Natural Resources, which will give further emphasis to this important subject, and serves to show how thoroughly chemists are alive to the subjects considered by this Congress.

The American Electrochemical Society, representing in its membership large users of power for electrochemical processes, approves the following statement of "Some Principles of Water Conservation," and recommend it for the consideration of the Third National Conservation Congress about to be held at Kansas City, Mo., September 25, 26, and 27, 1911.

SOME PRINCIPLES OF WATER CONSERVATION.

First. True conservation should begin with the headwaters of streams. Forestry and impounding waters in ponds and lakes should go hand in hand.

Second. Farmers should be encouraged to develop the water power from their farm brooks.

Third. Each State should set apart a fund to aid farmers in making a safe and scientific development of water for storage, power and irrigation purposes, to be returned to the fund in easy installments to be again used in helping others in the same way.

Fourth. It is advised that to render such farm developments scientific and safe, each State should employ a competent engineer body, who should plan and take charge of such constructions for their harmonious development in connection with other developments in the same water system, feeding the excess production of power or water for irrigation into a common system, so that every feature be as fully utilized as possible.

Fifth. Each State should develop at least one river system to its utmost capacity, including minor streams for storage, power and irrigation as an object lesson to its own people and those of other States.

Sixth. It is preferable that a navigable river be taken for such demonstration and developed to its utmost capacity in forestry, water storage, irrigation, power navigation and the reclamation of valuable flooded lands in its lower levels, each interest co-ordinated to make a complete whole, the excess of power generated being fed into a common system and made available for use.

Seventh. No navigable stream that has water power possibilities of value as well as navigation, should be developed exclusively and only for navigation purposes.

Eighth. No water power streams that can well be co-ordinated to a profit for the purpose of navigation should be developed exclusively as a water power stream.

Ninth. Streams having low lands of great value subject to overflow by floods should be investigated in their head waters with reference to forestry and water storage therein, making comparative estimates of the

value of lands required for storage and forestry with those that would be thereby saved from overflow and turned to greater utility.

Tenth. If such investigation should justify, it should be made possible to acquire such necessary lands for forestry or storage upon an equitable basis, even if very remote from the lands to be benefited and to assess the expense upon the lands so benefited to be paid in easy annual payments to a fund usable again for similar purposes.

Eleventh. The several States should enter into some suitable inter-State agreements which would enable a State having large areas of valuable land subject to overflow from rivers having their origin in other States by which necessary storage areas could be made available for such purpose, an equity of compensation being made for areas set apart for such use.

Twelfth. The funds thus made available from another State should be in charge of such State's Water Supply Commission or the equivalent to aid in its development of water storage in connection with its own system of water power developments.

Suitable laws being enacted by the several States providing for such compensated use of lands in a remote State.

If such investigation should justify it should be made possible to acquire such necessary lands for forestry or storage upon an equitable basis, even if remote from the land benefited and assess the expense upon the land so benefited in an equitable way.

It is also advised that hydraulic, chemical and electrical engineers be included with other engineers in the solution of conservation problems.

In the evening a demonstration run was made in the Electric Furnace Laboratory of the Chemistry and Mining Building of the Evans electric furnace, followed later on by a smoker.

During the afternoon the ladies in attendance motored through the city, followed by a theatre party in the evening.

SESSION OF SEPTEMBER 22d.

The session was called to order at 10 A. M. at the beautiful home of the Lambton Golf and Country Club, President W. R. Whitney in the Chair.

Papers by S. Dushman, Alcan Hirsch, I. Langmuir, C. F. Hale (read in abstract by President Whitney), J. W. Richards, Alfred Sang (read by title) were presented and discussed, as printed in full in these Transactions.

Mr. E. R. Taylor then moved, Mr. Carl Hering seconding, the following:

Motion of Thanks.

That the thanks of the American Electrochemical Society be extended to:

The President of the University of Toronto;

The Chairman of the Canadian Section of the Society of Chemical Industry;

The General Committee, W. L. Miller, Chairman, and W. P. Cohoe, Secretary;

The Ladies Committee;

Lt. Col. and Mrs. Miller;

Mr. J. W. Evans;

The Lambton Golf and Country Club;

The Canadian Westinghouse Company;

The Steel Company of Canada;

The Hydro-Electric Power Commission of Ontario.

The motion was carried unanimously.

The afternoon was given over to recreation, golf, and a reception by Lt. Col. and Mrs. Miller at their residence.

In the evening dinner was held at McConkey's restaurant.

Special cars attached to the Grand Trunk train carried members and guests to Hamilton, where visits were made to the works of The Canadian Westinghouse Company, The Steel Company of Canada and the transformer and testing station of the Hydro-Electric Commission's 110,000 volt transmission at Dundas.

MEMBERS AND GUESTS REGISTERED AT THE TWENTIETH GENERAL MEETING.

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T. F. Baily	S. L. Bigelow	J. F. Callahan
J. Watson Bain	Wm. Brady	H. R. Carveth

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E. R. Cole
Wm. M. Corse
J. S. Crider
Saul Dushman
J. W. Evans
F. A. J. FitzGerald
M. W. Franklin
R. H. Gaines
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F. A. Stamps
Edw. R. Taylor
Robert Turnbull
L. D. Vorce
Oliver P. Watts
R. J. Weitlaner
W. R. Whitney
A. M. Williamson

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Mrs. F. A. J. FitzGerald
Miss Mabel E. Gibbs
Miss M. von Isakovics
Mrs. Kelleher
Miss Kelleher
Mrs. E. F. Northrup
Mrs. A. G. Rodgers
Mrs. Edw. R. Taylor
Mrs. L. D. Vorce
Mrs. Oliver P. Watts

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A. Nieghorn
A. C. Parsons
Jos. Race
C. H. Rich
T. Somery
W. C. Stone
R. W. Thornson
G. B. Waterhouse
Roy Winne

Members Elected and Qualified.

April, 1911—September, 1911.

April 29, 1911.

Frank McDonald, Roaring Springs, Pa.
Earl Blough, Parnassus, Pa.
Victor Doerschuk, Niagara Falls, N. Y.
Jas. Wm. Moffat, Toronto, Canada.
Seth A. Moulton, Portland, Me.
John Collins Clancy, New York City.
Henry B. Haigh, New York City. Res., Philadelphia, Pa.
Arthur E. Hall, Omaha, Neb.
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Dr. E. A. Beck, New York City.
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J M. Camp, Duquesne, Pa.
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Clarence S. Lomax, New York City.
Samuel Peacock, Baltimore, Md.
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James N. Pearce, Iowa City, Iowa.
John C. Reed, Steelton, Pa.
N. V. Hansell, New York City.
Matthew A. Hunter, Troy, N. Y.
L. Bertram Schleeder, New York City.
Max Gustav Weber, Dr., Ing., Perth Amboy, N. J.
Stephen R. Morey, New York City.
Frank O. Clements, Dayton, Ohio.
Geo. Wm. Sargent, Pittsburgh, Pa.
William F. Rockwell, Gary, Ind.
H. F. McCullough, Niagara Falls, N. Y.
Paul Arnemann, Hohenlohehuette, Upper Silesia, Germany.
Dr. Jingoro Yamasaki, Yonezawa, Japan.

May 27, 1911.

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Willie H. Baltzell, Pittsburgh, Pa.
Prof. Alfred H. Bucherer, Bonn, Germany.
Reinhard Beutner, New York City.
O. H. Wurster, Cincinnati, Ohio.
Harold van der Linde, New York City.
Wm. W. Clark, Bridgeville, Pa.
Oliver O. App, New York City, N. Y.
Dorsey E. McLaughlin, Seattle, Wash.
Wm. H. Connell, Pittsburgh, Pa.
Christopher A. Russell, Niagara Falls, N. Y.
Thos. R. Ernest, Spokane, Wash.
Henry S. Amer, Bloomfield, N. J.
Frederick J. Hambly, Buckingham, Quebec, Canada.
C. F. Elwell, Palo Alto, Cal.
Wm. Chas. Bauer, Evanston, Ill.
Francis D. Weeks, New York City.

June 21, 1911.

Charles Ferry, Bridgeport, Conn.
Jens Orten-Boving, London, E. C., England.
George D. Van Arsdale, New York City.
Zach Webb, Pittsburgh, Pa.
Wm. B. Waldo, New York City.
John B. Taylor, Schenectady, N. Y.
Dr. Alois Helfenstein, Vienna, XIX, Austria.
Ching Yu Wen, New York City.

Members Elected and Qualified (Continued).

July 21, 1911.

Albert A. Heimrod, Newark, N. J.
C R. Corning, New York City.
Frederic S. Mason, M. D., New York City.
Irving R. Valentine, Schenectady, N. Y.
Thomas Duncan Robertson, Toronto, Canada.
Archibald W. Diack, Detroit, Mich.
Chas. R. Barton, Ellwood City, Pa.

August 25, 1911.

Thos. R. Loudon, Toronto, Can.
Frederick Gelstharp, Creighton, Pa.
Axel Hartman, Tyssedal, Hardanger, Norway.
Albert V. Bleininger, Urbana, Ill.
Philip H. Falter, Shawinigan Falls, Que., Canada.
Walter N. Crafts, Cleveland, Ohio.
Harald Lundgren, Chicago, Ill.
Wm. D. Mason, Pt. Richmond, Cal.

September 20, 1911.

Alan Kissock, Golden, Col.
Ervin J. Weber, Madison, Wis.
Joseph F. Callahan, Niagara Falls, N. Y.
Preston W. Holtzendorff, Memphis, Tenn.
Royal E. Frickey, Redding, Cal.
Haydn Cornthwaite, Sogndal i. Dalene, Norway.

AMERICAN ELECTROCHEMICAL SOCIETY.

DIRECTORY OF MEMBERS.

November 1, 1911.

(*Members of date April 3, 1902, are charter members*)

ABADIE, Eugene H. (Sept. 26, '08) Consult. Eng., 1720 Third Nat. Bank Bldg., St. Louis, Mo.; res., 5148 Westminster Place.
ABBE, Paul O. (Nov. 27, '09) res., 4434 18th Ave., Brooklyn, N. Y.
ABBOTT, William G. Jr. (Mar. 27, '09) care of Hillsboro Mills, Wilton, N. H.
ACHESON, E. G. (Apr. 3, '02) Inter. Acheson Graphite Co., Niagara Falls, N. Y.
ACHESON, Edward G., Jr. (July 31, '08) Pres. Acheson Oildag Co., Niagara Falls, N. Y.
ACKER, Chas. E. (Apr. 3, '02) care of Secretary.
ADAMS, Prof. C. A. (Apr. 3, '02) Asst. Prof. of Elec. Eng., Harvard University, Cambridge, Mass.
ADAMS, Louis W. (Feb. 25, '11) Asst. Supt., Saucon Plant, Bethlehem Steel Co., South Bethlehem, Pa. Res., 844 Delaware Ave.
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BEIGNET, Raymond (Feb. 27, '09) Chemist, Compania Minera dos Estrellas, El Oro., Mexico; Apartado No. 34.
BENECKE, Adelbert O. (Sept. 26, '08) care of Industrial Instrument Co., Foxboro, Mass.
BENNETT, Benjamin F. (Feb. 26, 10) c/o American Sheet and Tin Plate Co., Vandergrift, Pa.
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BENNIE, P. McN. (July 1, '04) FitzGerald & Bennie Laboratories, Niagara Falls, N. Y.
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BERGSCHOLD, Iven A. J. (Nov. 26, '10) Electrometallurgical Engr., A. B. Electrometall, Trollhattan, Sweden.
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BIERBAUM, Christopher H. (Apr. 3, '02) Consult. Eng., Vice-Pres. Lumen Bearing Co., Pres. Robson Smelting Co., 418 Prudential Bldg., Buffalo, N. Y.
BIGELOW, S. Lawrence, Ph. D. (May 9, '03) Prof. of Gen. and Phys. Chem., Univ. of Mich.; res., 1520 Hill St., Ann Arbor, Mich.
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BLAKE, Lucien I., Ph. D. (Mar. 5, '04) Submarine Signal Co., 1936 Curtis St., Denver, Colo.
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BONNA, Dr. Aug. E. (Apr. 2, '04) Rue Petitot 15, Geneve, Switzerland.
BOON, Prof. John D. (Apr. 3, '02) Polytechnic College, Fort Worth, Tex.
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BOYNTON, Arthur J. (Feb. 26, '10) Superintendent Blast Furnaces, The National Tube Co., Lorain, Ohio; res., 230 Third St., Elyria, Ohio.
BRADFORD, Robert H. (Feb. 25, '11) Professor of Metallurgy, University of Utah, Salt Lake City, Utah.
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BRADLEY, James H. (June 25, '10) Chief Electrician, Jones & Laughlin Steel Co., Pittsburgh, Pa.; res., 858 Killey Moon St.
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Loeb, M.
Love, E. G.
Lovejoy, D. R.
Lucke, H. J.
MacDonald, J. A.
Mailloux, C. O.
Martin, T. C.
Mason, E. J. K.
Mason, F. S.
Mastick, S. C.
Mayer, F. J.
Maynard, G. W.
Mays, E. A.
McAllister, A. S.
McBerty, F. R.
McKenna, C. F.
McMurtrie, W.
McNeill, R.
Mershon, R. D.
Merton, R.
Metz, H. A.
Monrath, G.
Moody, H. R.
Morey, S. R.
Morgan, J. L. R.
Morrow, J. T.
Morse, W. S.
Muir, J. M.
Muschenheim, F. A.
Nicholas, F. C.
Nichols, W. H.
Nichols, W. S.
Nodell, W. L.
Parker, H. C.
Pattison, F. A.
Pearson, F. S.
Pennie, J. C.
Perry, R. P.
Price, E. F.
Prindle, E. J.
Prosser, H. A.
Randolph, E.
Reber, S.
Riglander, M. M
Riker, J. J.
Robb, C. D.
Roeber, E. F.
Roller, F. W.
Rossi, A. J.
Rowland, L.
Rudra, S. C.
Ruhl, L.
Rynard, W. T.
Schloss, J. A.
Schuetz, F. F.
Seward, G. O.
Sharp, C. H.
Skowronski, S.
Smith, E. A. C.
Speiden, C. C.
Spice, R.
Spilsbury, E. G.
Stanton, F. McM.
Stevens, O. E.
Stoll, C. G.
Stone, I. F.
Stoughton, B.
Stovel, R. W.
Strong, A. H.
Takamine, J.
Taylor, J. R.
Teeple, J. E.
Thatcher, C. J.
Thompson, J.
Thompson, R. N.
Tidd, G. N.
Toch, M.
Tucker, S. A.
Van Arsdale, G. D.
Van der Linde, H.
Waddell, M.
Waldo, L.
Waldo, W. B.
Walker, A. L.
Waterman, F. N.
Weaver, W. D.
Weeks, F. D.
Weinstein, L. J.
Wells, G. A.
Wen, Ching Yu
White, J. G.
Widmann, E. A.
Wiechmann, F. G.
Wigglesworth, H.
Williams, A.
Williams, J. T.
Winship, W. E.
Wittnebel, E. S.
Wolff, R. H.
Wood, E. F.
Worth, B. G., Jr.

Niagara Falls—

Acheson, E. G.
Acheson, E. G., Jr.
Allen, T. B.
Arison, W. H.
Barton, P. P.
Becket, F. M.
Beckman, J. W.
Bennie, P. McN.
Callahan, J. F.
Carveth, H. R.
Cole, E. R.
Converse, V. G.
Cox, G. E.
Doerschuk, V. C.
Dunlap, O. E.
Edmands, I. R.
FitzGerald, F. A. J.
Fowler, R. E.
Furness, G. C.
Giles, I. K.

Greene, F. V.
Hall, C. M.
Hall, S. F.
Harper, J. L.
Haskell, F. W.
Hepburn, D. M.
Herzog, G. K.
Horry, W. S.
Hooker, A. H.
Imlay, L. E.
Kellogg, H. W.
Koethen, F. L.
Lidbury, F. A.
MacMahon, J.
Marseilles, W. P.
Marsh, C. W.
Marshall, J. G.
Mauran, M.
McCullough, H. F.
Meredith, W. F.
Moritz, C. H.
Moyer, G. C.
Ornstein, G.
Petitnot, N.
Phillips, R.
Reeve, A. G.
Rodgers, A. G.
Ross, Wm.
Russell, C. A.
Saunders, L. E.
Sergeant, E. M.
Simmers, A. L.
Smith, E. S.
Smith, W. A.
Snowdon, R. C.
Speiden, E. C.
Sprague, E. C.
Spurge, E. C.
Stamps, F. A.
Stone, G. W.
Taylor, Chas. E.
Tone, F. J.
Vaughn, C. F.
Wallace, W.
Wilson, I. W.
Wilson, J. R.
Williamson, A. M.
Yoe, Harry A.

North Tonawanda—
Randall, J. W. H.

Penn Yan—
Taylor, E. R.

Piermont—
Main, W.

Poughkeepsie—
Moulton, C. W.

Prince Bay, S. I.—
Hammond, J. F.
Johnston, F. A.
Johnston, W. A.

Richmond Hill, L. I.—
Haslwanter, C.
Maeulen, F.

Rochester—
Hutchings, J. T.
Parker, J. C.
Schwarz, R. C.

Schenectady—
Andrews. W. S.
Arsem, W. C.
Bartlett, F. S.
Brunt, Van, C.
Capp. J. A.
Creighton, E. E.
Diller, H. E.
Franklin, M. W.
Hansen, C. A.
Langmuir, I.
Murphy, E. J.
Reist, H. G.
Rice, E. T.
Rushmore, D. B.
Seede, J. A.
Steinmetz, C. P.
Taylor, J. B.
Valentine, I. R.
Whitney, W. R.
Willey, L. M.

Syracuse—
Brookfield, W. B.
Matthews, J. A.
Pennock, J. D.
Scott, W. H.
Sykes, H. W.

Troy—
Betts, A. G.
Hunter, M. A.
Lincoln, A. T.
Robb, W. L.

Utica—
Johnson, M. H.
Schiller, F. W.

West New Brighton, S. I.—
Ingalls, W. R.

Yonkers—
Baekeland, L.
Brown, J. S.
Harrington, E. I.
Statham, Noel

NORTH CAROLINA.

Chapel Hill—
Patterson, A. H.

Charlotte—
Gilchrist, P. S.

West Raleigh—
Browne, W. H.

NORTH DAKOTA.

Agricultural College—
Dolt, Maurice L.

OHIO.

Akron—
Geer, W. C.
Shaw, E. C.

Alliance—
Baily, T. F.

Ashtabula—
Harrar, E. S.

Cleveland—
Brady, W. B.
Brown, J. W.
Chillas, R. B., Jr.
Clymer, W. R.
Collins, 2d, C. L.
Crafts, W. N.
Crider, J. S.
Fleming, R.
Fleming, S. H.
Glascock, B. L.
Graves, W. G.
Hill, Chas. W.
Hobbs, P. L.
Hudson, A. J.
Humel, Edward J.
Hyde, E. P.

Koehler, W.
Lane, H. M.
Mann, W. W.
Mott, W. R.
Ordway, D. L.
Pritz, W. B.
Pulman, O. S.
Seabury, R. L.
Smith, A. W.
Snyder, J. L. K.
White, F. L.
Whitlock, E. H.
Woodward, J. M.

Columbus—
Allensworth, H. R.
Barnes, A. H.
Withrow, J. R.

Dayton—
Clements, F. O.
Cridland, H. C.
Deeds, E. A.
Niswonger, E. E.

Elyria—
Boynton, A. J.
Briggs, F. H.

Fostoria—
Downes, A. C.

Fremont—
Goodwin, J. H.
Ziegler, W. W.

Lakewood—
Brown, J. W.
Humel, E. J.
Ordway, D. L.
Pulman, O. S.
White, F. L.

Lorain—
Boynton, A. J.

Middletown—
Ahlbrandt, G. F.
Richardson, E. A.

Oberlin—
Crafts, W. N.

Salem—
Davis, D. L.

Steubenville—
Homan, J. G.

Youngstown—
Brinker, H. L.

OREGON.

Eugene—
Shinn, F. L.

PENNSYLVANIA.

Aliquippa—
Stafford, S. G.

Altoona—
Casselberry, H.
Ohlwiler, C. H.
Walters, H. E.

Aspinwall—
Hessom, B. F., Jr.

Bethlehem—
Landis, W. S.

Brackenridge—
Connell, H. R.

Braddock—
Friedlaender, E.
Lewis, J. D.

Bridgeville—
Clark, W. W.
Saklatwalla, B. D.

Bryn Mawr—
Ely, T. N.

Canonsburg—
Danforth, C. W.

Canton—
Taylor, F. D.

Chester—
Comey, A. M.

Coatesville—
Coates, J.

Colwyn—
McConnell, J. Y.

Coraopolis—
Stafford, S. G.

Creighton—
Gelstharp, F.
Kerr, Chas. H.

Duquesne—
Camp, J. M.
Cornell, S.
Reed, R. C.
Unger, J. S.

Easton—
Adamson, G. P.
Gordon, C. McC.
Hart, E.
Shimer, E. B.

East Pittsburgh—
Gibson, C. B.

Economy—
Storey, O. W.

Edgewood—
Donkin, Wm. A.

Ellwood City—
Barton, Chas. R.
Dunn, J. J.

Gettysburg—
Parsons, L. A.

Ingram—
Armor, J. C.

Johnstown—
Kenney, E. F.
Parkhurst, C. W.

Lancaster—
Schwarz, A.

Latrobe—
Eckfeldt, J. J.

Lebanon—
Crowell, W. J., Jr.
Weimer, E. A.

McKeesport—
Clarke, E. B.
Farnham, F. F.
Goodspeed, G. M.
Hensen, Emil

Moore—
Werliin, G.

Munhall—
Hartzell, L. M.
Hopkins, G. A.
James. R.
McNiff, G. P.
Reese, P. P.
Stevens, R. H.

Natrona—
White, R. H.

Norwood—
Goldbaum, J. S.

Palmerton—
Hendricks, W. H.
Koch, S. B.
Smull, J. G.

Parnassus—
Blough, Earl

Philadelphia—
Bacon, E. W.
Bower, F. B.
Breed, G.
Cary, C. R.
Chalas, Adolphe
Chance, H. M.
Clamer, G. H.
Devereux, W.
Eglin, W. C. L.
Fahrig, E.
Flagg, S. G., Jr.
Fraley, J. C.
Furness, R.
Hering, C.
Hess, H.
Hicks, Edwin F.
Hitchcock, F. R. M.
Hornor, H. A.
Howard, G. M.
Keith, N. S.
Kitsee, I.
Lafore, J. A.
Lavino, E. J.
Lay, J. Tracy
McConnell, J. Y.
McFarlin, J. R.
Meyer, J.
Morris, H. G.
Paul, H. N.
Perry, R. S.
Queneau, A. L.
Reed, C. J.
Rosengarten, G. D.
Russell, C. J.
Sadtler, S. P.
Sadtler, S. S.
Salom, P. G.
Schamberg, M.
Smith, E. F.
Smith, E. W.
Snook, H. C.
Steinmetz, J. A.
Stevens, J. F.
Taggart, W. T.
Thwing, C. B.
Weeks, C. A.
Werliin, Godwin
White, A. J.
Wickes, C. S.
Williams, H. J.

Pittsburgh—
Amberg, Richard
Babb, Jos. E.
Baltzell, W. H.
Bradley, Jas. H.
Brown, G. H.
Brown, John T., Jr.
Connell, W. H.
Crabtree, F.
Davis, S. A.
Dewey, B.
Donkin, Wm. A.
Dougherty, J. W.
Edwards, Albert D.
Fisher, Henry W.
Flannery, Jas. J.
Goodale, S. L.
Graves, Jas. M.
Hartley, R. H.
Hessom, B. F., Jr.
James, J. H.
Kelley, O. C.
Kemery, P.
Kier, S. M.
Laughlin, H. H.
Lincoln, P. M.
McDonald, R. A.
McKinley, Jos.
McKirdy, John E.
Merrill, J. L.
Messler, E. L.
Mueller, H. N.
Murphy, D. H.
Ober, J. E.
Orr, R. S.
Osborne, L. A.
Page, G. S.
Pinkerton, A.
Pope, Chas. E.
Rodman, H.
Saklatwalla, B. D.
Sargent, G. W.
Schluederberg, C. G.
Scott, C. F.
Sill, H. F.
Slocum, Chas. V.
Slocum, F. L.
Snelling W. O.
Speller, F. N.
Stewart, R. T.
Sturgess, F. M.
Uhlenhaut, F., Jr.
Vogt, C. C.
Webb, Z.
Wiley, B.
Yardley, J. L.

Pottstown—
Coleman, W. B.
Cook, E. B.

Pottsville—
Chance, E. M.

Primos—
Boericke, G.
Stein, W. M.

Reading—
Parker, Jas. H.

Roaring Springs—
McDonald, F.

Saltsburg—
Kelley, O. C.

Scranton—
Bryden, C. L.

Sewickley—
Wadsworth, F. L.

Sharon—
Kranz, W. G.
Samuels, W. P.
Warren, G. S.

Sinnamahoning—
Gray, C. W.

South Bethlehem—
Acker, C. E.
Adams, L. W.
Buck, C. A.
Franklin, W. S.
MacNutt, B.
Richards, J. W.
Seyfert, S. S.

State College—
Pond, G. G.
Richardson, H. K.
Taylor, F. D.
Turnock, L. C.
Walter, C. A.

Steelton—
Calder, A. R.
Carney, F. D.
Lindemuth, L. B.
Reed, J. C.

Swarthmore—
Alleman, G.
Swissvale—
Sturgess, F. M.
Tarentum—
Connell, H. R.
Hitchcock, H. K.
Treichlers—
Lentz, H. N.
Tyrone—
Bachert, A. E.
Vandergrift—
Bennett, B. F.
Washington—
McAdam, D. J., Jr.
West Pittsburgh—
Buch, N. W.
Wilkinsburg—
Gibson, Chas. B.
Hillegass, H. H.
Skinner, Chas. E.
York—
Schleeder, L. B.

RHODE ISLAND.

Greene—
Perry, C. M.
Newport—
Richards, E.
Providence—
Catlin, C. A.
Saunders, W. M.

SOUTH CAROLINA.

Columbia—
Mills, J. E.
Sumter—
Deventer, Van, H. R.

TENNESSEE.

Chattanooga—
Thomas, B. F.
Clarksville—
Faust, T. B.
Memphis—
Holtzendorff,, P. W.
Nashville—
Dudley, W. L.
Washburn, F. S.

TEXAS.

Austin—
Harper, H. W.
Schoch, E. P.
Fort Worth—
Boon, J. D.
Port Arthur—
Slater, W. A.

UTAH.

Provo City—
Smith, F. W.
Salt Lake City—
Bradford, R. H.
Ernest, T. R.
Jennings, E. P.
Merrill, J. F.

VIRGINIA.

Holcomb's Rock—
Kugelgen, F. von
Lee, H. R.
Norfolk—
Webb, L. W.
Roanoke—
Engle, H. M.
University—
Dunnington, F. P.

WASHINGTON.

Irondale—
Merzbacher, A.
Seattle—
Crim, L. P.
McLaughlin, D. E.
Spokane—
Armstrong, L. K.
Ernest, T. R.
Woodwell, A. H.
Tacoma—
Brandt, O. T. D.
Everette, W. E.

WEST VIRGINIA.

Clarkesburg—
Chorpening, G. B.
Glen Ferris—
Huffard, J. B.
Morgantown—
Hite, B. H.
Roush, Gar A.
Wheeling—
Hearne, D. G.

WISCONSIN.

Eau Claire—
Peirce, A. E.
Madison—
Aston, J.
Beebe, M. C.
Bennett, E.
Burgess, C. F.
Burling, B. B.
Gorman, H. B. L.
Hambuechen, C.
Kahlenberg, L.
Kowalke, O. L.
Schulte, W. B.
Watts, O. P.
Weber, E. G.
White, A. R.
Marinette—
Lathrop, L. H.
Milwaukee—
Kremers, J. G.
Lord, Chas. E.
Theurer, F. L.
Vogel, G. C.
Woodbury, F. E.
Zimmermann, J. G.
Wauwatosa—
Tifft, G. L.

WYOMING.

Thermopolis—
Freeman, G. N.

CANADA.

British Columbia—
Ashcroft—
Bales, H. H.
Heriot Bay—
Bagot, C. G.
Greenwood—
Keffer, F.
Nelson—
Fowler, S. S.

Vancouver—
Barwick, W. S.
Boesch, J. E.
Hayward, R. F.
Kendall, G. R.
Manitoba,
Winnipeg—
Morden, G. W.
Nova Scotia,
New Glascow—
Cantley, T.
Sydney—
Hitt, S. E.
Scott, A. P.
Ontario,
Cobalt—
Evans, J. W.
Copper Cliff—
Browne, D. H.
Fitzroy Harbor—
Simpson, L.
Gowganda—
Colvocoresses, Geo. M.
Kingston—
Goodwin, W. L.
Guttman, L. F.
Niagara Falls—
Cooper, K. F.
Knorr, A. E.
Port Arthur—
Hille, F.
Sault Ste. Marie—
Sjostedt, E. A.
Smithrim, E. R.
Soskatoon—
MacLaurin, R. D.
St. Catharines—
Heitmann, E.
Toronto—
Cohoe, W. P.
Dushman, S.
Irving, T. C., Jr.
Kaemmerer, J. A.
Kirkegaard, P.
Loudon, T. R.
Miller, W. L.
Moffat, J. W.
Patch, N. K. B.
Robertson, T. D.
Tate, Alfred O.
Watts, Geo. W.
Wurster, O. H.
Welland—
Turnbull, R.
Quebec,
Buckingham—
Hambly, F. J.
Montreal—
Hough, A.
McIntosh, D.
Stansfield, A.
Walker, J. W.
Ottawa—
Haanel, E.
Haff, M.
Morgan, L. C.
Shawenigan Falls—
Falter, P. H.
Witherspoon, R. A.

COSTA RICA.

Puntaremus—
Fraser, Lee

CUBA.

Isle of Pines—
Roberts, I. L.
Santiago de Cuba—
Bruns, C. L., Jr.

MEXICO.

El Oro—
Beignet, R.
Girdwood, K. J.
La Boquilla—
Hobble, A. C.
Mexico City—
Brindley, Geo. F.
Ferguson, W. A.
Vrooman, H. H.
Wilkins, E. M.
Teguisguiapan—
Honey, W.
Tezuitlan—
Watson, Ralph A.
Torreon—
Mills, H. P.

NEWFOUNDLAND.

St. John's—
Harvey, E. F.

PANAMA.

Culebra—
Schildhauer, E.

SOUTH AMERICA.

ARGENTINE REPUBLIC.

Buenos Aires—
Newbery, Jorge
Stone, J. P.

BRAZIL.

Rio de Janeiro—
De Medeiros, T. S. V.
Guinle, Eduardo
S. Paulo—
De Souza, E.
Walmsley, W. N.
Villa Nova de Lima—
Gill, T. P.
Jones, H.

CHILI.

Antofagasta—
Atwood, C. E.
Santiago—
Blanquier, J. T.
Diaz-Ossa, B.

COLOMBIA.

Bogota—
Calvo, P. P.
Pasto—
Gamba, F. P.

PERU.

Cerro de Pasco—
Guess, Geo. A.
Lima—
D'Ornellas, T. V.
Holstein, O.

AUSTRALASIA.

NEW SOUTH WALES.

Broken Hill—
Gepp, H. W.

Port Kembla—
Johns M. J.
Lawson, J. L.
Magnus, B.
White, E. A.

Moonta Mines—
Hancock, H. L.

Sydney—
Corin, W.
May, Alex.

Waratah—
Schroeder, H.

NEW ZEALAND.

Dunedin—
Creagh, E. C.
Stark, E. E.

Epsom—
Gordon, H. A.

Wellington—
Fahy, J. T.

QUEENSLAND.

Brisbane—
Jackson, A. G.

Mt. Morgan—
Du Faur, J. B.
Richard, G. A.

TASMANIA.

Queenstown—
Sticht, R. C.

AFRICA.

CAPE COLONY.

Somerset, West—Quinan, K. B.

ASIA.

CHINA.

Lin-sien—
Kwong, Y. K.

INDIA.

Bangalore—
Arunachela, T. K.

Rejouli—
Dickson, A. A. C.

Kankanhalli—
Tate, J. V. E.

JAPAN.

Kure—
Tada, S.

Kyoto—
Namba, M.
Nishikawa, K.
Yoshikawa, K.

Naganoken—
Takahashi, T.

Takata—
Kunitomo, S.

Tokyo—
Hoshina, T.
Iwai, K.
Katsura, B.
Kishi, K.
Masujima, B.
Nohara, T.
Oshima, Y.

Yonezawa—
Yamasaki, J.

PHILIPPINE ISLANDS.

Manila—
Dalburg, F. A.

EUROPE.

AUSTRIA-HUNGARY.

Prague—
Babrowsky, G.

Vienna—
Helfenstein, A.
Paweck, H.

BELGIUM.

Brussels—
Moltkehansen, I. J.
Solvay, A.

FINLAND.

Helsingfors—
Oholm, L. W.

FRANCE.

Paris—
Drake, F. E.
Gall, H.
Garfield, A. S.
Gin, G.
Keller, C. A.
Marie, C. A.
Ostheimer, J. W.
Riva-Berni, A.

Saint-Cloud—
Sang, A.

GERMANY.

Aachen—
Classen, A.

Berlin—
Glenck, I.
Reichel, W.

Bonn—
Bucherer, A. H.

Charlottenburg—
Engelhardt, V.
Fels, B.

Clausthal—
Hoffman, R.

Dresden—
Schrey, A.

Essen-Ruhr—
Goldschmidt, H.

Frankfurt—
De Neufville, R.
Liebmann, L.
Hamburg—
Wohlwill, H.
Hanau—
Heraeus, H.
Hoechst—
Reisenegger, H.
Hohenlohehuette—
Arnemann, P.
Karlsruhe—
Askenasy, P.
Haber, F.
Leipzig—
Le Blanc, M.
Schall, C.
Wagner, J.
Linz—
Calberla, R.
Lubeck—
Draeger, A. B.
Werdingen—
Meyer, F.

GREAT BRITAIN.

Birmingham—
Smith, C. A.
Threlfall, R.
Cardiff—
Ewing, A. J.
Letchworth—
Foster, C. E.
Liverpool—
Smith, A. T.
London—
Ashcroft, E. A.
Boving, Jens O.
Campbell, D. F.
Cowper-Coles, S.
Merz, C. H.
Ruthenberg, M.
Shields, J.
Stütz, E.
Wilder, F. L.
Manchester—
Pring, J. N.
Rugby—
Frodsham, H.
Sheffield—
Fawcett, P.
Hadfield, R. A.
Hutton, R. S.
Rowland, T.
St. Helens—
Le Mare, E. B.
West Ealing—
Irvine, H. A.

ITALY.

Aguila—
Dapples, A.
Rome—
Catani, R.
Chiaraviglio, D.
Morani, F.
Turin—
Giolitti, F.
Stassano, E.

NORWAY.

Bergen—
Irgens, J. F.
Petersson, A.
Christiania—
Bjornson, E.
Hiorth, A.
Hiorth, F. V. L.
Johansen, G. H.
Styri, H.
Jossingford—
Borgen, A.
Cornthwaite, H.
Cornthwaite, S.
Odda—
Olsen, H.
Ski—
Soederberg, C. W.
Stavanger—
Jacobsen, F.
Trondheim—
Tharaldsen, F.
Tyssedal—
Hartman, A.

RUSSIA.

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SPAIN.

Rio Tinto—
Shields, J.

SWEDEN.

Falun—
Ljungh, H.
Yngstrom, L.
Gullspang—
Hedlund, M.
Hoganas—
Akerberg, O. T.
Forssell, J.
Ludvika—
Groenwall, Assar
Lindblad, Axel
Stalhane, Otto
Mansbo—
Carlson, B.
Stockholm—
Appelberg, A. O.
Leffler, J. A.
Tingberg, Otto
Trollhattan—
Bergschold, I.
Cornelius, E.
Olausson, K. O. E.
Vesteras—
Edstrom, J. S.
Westervik—
Tillberg, E. W.

SWITZERLAND.

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Gandillon, A.
Guye, P. A.
Lacroix, H.
Turgi—
Landolt, H.
Zurich—
Liebmann, A. J.
Voge, A. L.

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THE PREPARATION AND PROPERTIES OF METALLIC CERIUM.*

By Alcan Hirsch.

INTRODUCTION.

In view of the amount of research[1] on the rare-earths, it is remarkable that little has been done on the metals themselves. This is easily understood, however, when one considers the scarcity of pure material to work with and the difficulty of reducing these very electropositive elements from their compounds. What metal has been obtained has been, in most cases at least, small in amount and very impure, usually contaminated with the other members of the group. On account of the rarity of the materials to work with, little has been done in a study of the physical and chemical properties of these rare-earth metals, and the field of alloys has been almost untouched.

In the present research, cerium, the leading member of the cerium group, was chosen as the rare-earth element to be studied. The investigation was undertaken with a two-fold purpose. First, starting from as pure cerium salts as it was possible to obtain, it was desired to prepare the metal in quantities sufficiently large that its physical and chemical properties could be studied and a large number of alloys could be made. Second, by preparing metal from the unpurified rare-earth residues from monazite sand, obtained as a by-product in the incandescent gas-mantle industry, it was hoped that alloys of commercial value could be obtained. In other words, whereas the application of these residues as oxalates or oxides is very limited to-day, their value might possibly be increased materially if a useful metallic or alloyed product be prepared from them.

The writer gratefully acknowledges his indebtedness to Pro-

* Presented as a thesis for the doctor's degree at the University of Wisconsin, June, 1911.

fessors Burgess and Lenher, of the University of Wisconsin, for the facilities of their laboratories and their helpful advice and encouragement during the progress of this research. The electrolytic work was done in the laboratories of Applied Electrochemistry, while the analyses and the chemical preparations were made in the Department of Inorganic Chemistry.

This research was begun in December, 1907, and concluded June, 1911. The actual time devoted to this study covered a period of over three years.

HISTORICAL.

In 1751 Cronstedt[2] discovered the mineral cerite in a mine of Bastnaës. In 1784 Bergmann and d'Elhyar[3] confused the rare-earths with lime, and published an incorrect analysis of this mineral cerite. In 1794 Gadolin[4] discovered the rare-earths in a heavy black mineral, afterwards called gadolinite.

In 1804 cerium was discovered simultaneously by Klaproth[5] in Germany and by Berzelius and Hisinger[6] in Sweden. The first investigator named the oxide ochre earth because of its color, but Berzelius and Hisinger called the new element cerium, after the planet Ceres. About the same time Vauquelin[7] confirmed the discovery of a new earth by analysing cerite, and then undertook the study of the salts of cerium.[8] In 1814 Berzelius and Galin[9] separated the oxides of cerium and yttrium. Between this period and that of the famous researches of Mosander, numerous articles were published on cerium and the minerals in which it was found, but the material used was necessarily very impure, and what was called at that time cerium oxide was in reality the mixed oxides of the cerium group.

In 1839 Mosander 10[10-11] announced that cerium oxide was a mixture of two oxides, those of cerium and lanthanum. In 1841 he stated that cerium oxide contained still another oxide, that of didymium. In 1868 Wolf[12] determined the equivalence of cerium and announced that the so-called cerium compounds were mixtures of two or more rare-earth elements. In 1879 Lecoq de Boisbandran,[13] while examining the didymia obtained from the mineral samarskite, isolated a new element which he called samarium. In 1885 Auer von Welsbach[14] showed that didymia

was a mixture of the oxides of two new rare-earth elements, neodymium and praseodymium.

At the present time there are about 15 well identified elements which, for chemical reasons, among other properties that of the insolubility of their oxalates in slightly acid solution, are classified as rare-earths. These rare-earths are usually subdivided into three groups in the order of their basicity—the cerium group, the gadolinium goup and the yttrium group. There is no absolute differentiation of one group from another, but rather a gradual transition. Usually only the five elements,—cerium, lanthanum, neodymium, praseodyium and samarium,—are included in the cerium group.

The present research is concerned with the cerium group, and principally with cerium itself. But in the rare-earths' relations between members of the same group are much closer than among other kindred elements, as the platinum metals. For example, the atomic weights of lanthanum, cerium and praseodymium are, respectively, 139, 140.25 and 140.6; therefore the chemistry of one element concerns that of the other members of the group,—to a certain degree at least.

Previous Work on the Rare-earth Metals.

Two general methods of preparation have been used for the production of the rare-earth metals:

(1) Reduction of the oxide or chloride by one of the very electropositive metals.

(2) Electrolysis of a fused salt.

Mosander and Marignac[15] appear to have been the first to produce metallic cerium by a thermal reduction using the alkali metals. Wöhler[16] describes the difficulty of the reduction using sodium metal and cerium chloride, and states that 12 grams of sodium yielded only 50-60 milligrams of cerium metal. Winckler[17] used metallic magnesium and cerium dioxide and obtained a pyrophoric mixture. Recently some attempts have been made to produce the metals by improved methods of thermal reduction. Matignon[18] used metallic aluminum and magnesium, and Schiffer,[19] in Muthmann's laboratory, experimented with the reduction of the oxides by aluminum. From the writer's own experiences in the attempted thermal reduction of these oxides,

which is described further on in this article, it appears that the reduction yields either a lower oxide, an impure mixture of partially reduced metal and oxide, or an alloy of the reduced metal and rare metal, such as an aluminum-cerium alloy.

In 1874 Frey[20] published a notice of the preparation of the rare-earth metals in the laboratory of Dr. Schuchardt, in Gorlitz, using the electrolytic method of Bunsen. The following year Hillebrand and Norton[21] published an account of their work on the electrolytic preparation of cerium. They succeeded in producing about six grams of cerium at a single operation, and obtained in all thirty grams of the metal. In 1895 Pettersson[22] attempted to electrolyze the oxides, but produced only the carbides. In 1902 Muthmann[23] and his associates published the first of a very comprehensive series of articles[24] on the metals of the cerium group. Besides preparing the metals of the cerium group, they devised a new type of electrolytic vessel which they found useful in their method of electrolysis. They have also prepared an alloy which they call "mischmetall," which is an alloy of the cerium-yttrium group metals. Borchers and Stockem[25] have devised a method in which they use the double cerium-calcium chlorides, and have patented this process (German Patent 172,529). The above-mentioned electrolytic methods will be referred to again further on in this article.

PREPARATION OF MATERIAL.

The general scheme followed in this research was to use the mixed rare-earth oxides for experimenting on the exact conditions required for the preparation of the desired salt, and after the proper conditions of the process had been determined, to prepare the pure cerium salt. Oxalates of the rare-earths, a by-product of the commercial extraction of thorium and cerium from monazite sand, were employed for these experiments. The oxides, prepared by calcining the oxalates at a temperature of 750-800° C. in a gas muffle furnace, analyzed 97.8 percent rare-earth oxides, of which approximately 49 percent was cerium dioxide. They dissolved completely in nitric acid, and also in hot concentrated hydrochloric acid on digestion, which is usually taken as a check that the cerium content is less than 50 percent.[26]

Preparation of Anhydrous Chlorides.

The first salts required were the anhydrous chlorides (composition RCl_3). There are several hydrated chlorides of cerium known, of the compositions: $2CeCl_3.15H_2O$; $CeCl_3.7H_2O$; $CeCl_3.6H_2O$; $CeCl_3.H_2O$. Cerium chloride is similar to magnesium chloride in that when its solution is evaporated to dryness and calcined, HCl is evolved with partial decomposition of the chloride to oxychloride and oxide.

A good many methods have been proposed for the preparation of anhydrous or dehydrated chlorides of cerium, among which may be mentioned the following: Heat the hydrated chlorides with NH_4Cl until the latter salt is completely volatilized.[27] Behringer[28] heated the hydrated chlorides with NH_4Cl in a glass tube in a current of chlorine. Robinson[29] heated the oxalates to a temperature of about 130° in dry HCl gas until all of the oxalic acid had sublimed, then gradually raised the temperature to a red heat. Didier[30] heated CeO_2 in a current of CO_2 and chlorine. Muthmann and Stutzel[31] passed H_2S over anhydrous sulphate heated to a high temperature, and then a mixture of CO_2 and dry hydrochloric acid gas. Mosander[32] burned cerium in chlorine. Moissan[33] attacked the carbide by hydrochloric acid gas at 650° or chlorine at 250°. Meyer[34] converted the oxides to chlorides by vapors of CCl_4.

A number of methods for the preparation of the anhydrous chlorides were tried before a suitable one was found. The large quantity of chloride which was required (several kilograms) necessitated that the preparation should be done in as simple and rapid a manner as was possible. Both aqueous and non-aqueous methods were tried, and below are given the different processes experimented with and their relative advantages and disadvantages.

One of the best methods for the preparation of this type of chlorides (those that decompose on evaporation and calcination from aqueous solution) is to heat the hydrated chloride with NH_4Cl. In the case of cerium chloride, Muthmann[35] describes in some detail this method, and mentions the tediousness and care required. To prepare a kilogram or so may require several days' work to effect the dehydration. The use of a platinum finger crucible is recommended, and not more than 50 grams of

the hydrated cerium-ammonium chloride should be heated at a time. The heating must be done very cautionsly or the product will be contaminated with oxychloride or oxide. The writer has tested thoroughly this method, and finds it very poor in this case for large scale operations. One set of experiments which was tried will illustrate the difficulties encountered. Two kilograms of the mixed oxides were dissolved in 4 liters HCl (22° Bé.) and about 4½ kilograms NH_4Cl were added. Evaporation was carried to dryness in large (35 cm.) porcelain evaporating dishes. About a kilogram of the chlorides was introduced into a large Dixon crucible, which was heated in a gas crucible furnace at a low temperature until the white fumes of NH_4Cl came off copiously. The fumes were conducted out of the window by a suitable flue, and the atmosphere within the furnace was at all times a reducing one. The heating was continued until the contents of the crucible were molten and white fumes ceased to come off. A strong odor of HCl gas and chlorine was noticed toward the end of the operation. The product did not melt to a clear liquid as the properly prepared anhydrous chloride does, but fused to a slimy mass. Analysis showed that the chloride was highly contaminated with oxide, and although 1,100 grams were obtained, the product was unsuitable for electrolysis.

Borchers and Stockem[36] state that many of the difficulties encountered in the dehydration of hydrated cerium chloride can be avoided by the preparation of the double calcium-cerium chloride, and that this salt can be prepared in the anhydrous state without decomposition by simple calcination. These results could not be duplicated by the writer. Forty-seven grams of the hydrated chloride of cerium were dissolved to a clear solution in water slightly acidified with HCl, and 15 grams of $CaCl_2$ were added. The solution was evaporated to dryness and calcined. The salt which was obtained did not melt to a clear solution, and was unsuited for electrolysis.

Dennis and Magee[37] have prepared the hydrated chloride $CeCl_3.7H_2O$, which loses water in a vacuum or over dehydrating agents, whereas the hydrate $2CeCl_3.15H_2O$ does not effloresce when placed over sulphuric acid.[38] It was thought that perhaps the anhydrous chloride could be prepared from the hydrated salt $CeCl_3.7H_2O$. The concentrated solution of the chlorides, pre-

pared by the action of concentrated HCl on the mixed oxides, was cooled to 0° C. by ice, and dry HCl gas was passed into the solution. After a time crystals began to separate out. They were filtered, dried in the air, and the chlorine content determined by titration with standard $AgNO_3$. The percent of chlorine was found to be 26.6 percent, whereas that in $CeCl_3.7H_2O$ is 28.6 percent. The presence of rare-earths other than cerium probably caused the difference in composition. When this salt was heated in a partial vacuum (water-pump) at 100° for two hours, the loss in weight was 4.4 percent. When dried over P_2O_5 in a vacuum desiccator for several days, the loss in weight was about 8 percent. The salt $CeCl_3.7H_2O$ was then dehydrated in dry HCl gas. Small quantities of the salt were placed in several porcelain boats in a porcelain tube furnace through which dry HCl gas was passed. The maximum temperature in the tube was 210°, and the average temperature was 195°. The product in the boat placed in the center of the tube lost 35 percent in weight, and analysis showed a chlorine content of 39.3 percent. The chlorine content of the anhydrous chloride $CeCl_3$ is 43.2 percent. The chloride so prepared melts to a clear liquid. Attempts were then made to prepare the anhydrous chloride on a larger scale by this method; 1,500 grams of the mixed oxides were converted into chlorides, and then the hydrate $CeCl_3.7H_2O$ was prepared by the method of Dennis and Magee, described above. The yield of salt was very small, and the operation is a difficult one to perform on a large scale, due to the fact that a low temperature is necessary and the absorption of HCl gas in water is accompanied by a considerable evolution of heat. Moreover, a simple method was found for preparing the monohydrate, and as the dehydration of the septa-hydrate passes through the stage of the monohydrate, this method was abandoned.

As the dehydration of the chlorides appeared to be the principal difficulty in the preparation of the anhydrous chlorides, it was thought that a suitable process might be developed by the use of non-aqueous solvents. As the action of non-aqueous solvents was found to be somewhat slower than the action of aqueous ones on cerium compounds, it was desirable that a

readily decomposable substance be used as the starting material, and the rare-earth carbides were chosen for this reason.

The carbide* RC_2 was prepared by heating the mixed oxides with powdered graphite in an arc furnace. Several kilograms of this material were prepared according to the method of Moissan.[39] The proper mixture to be heated was found to be 500 parts mixed oxides and 132 parts finest graphite powder. When a graphite crucible was used as one terminal and a graphite rod (about 1 inch diameter) as the other, the carbide was easily prepared on a large scale by using a 25 kw. arc (AC or DC). Care should be taken to protect the eyes properly, as the rays emitted from the hot mass are especially harmful as they contain a certain amount of ultra-violet rays.

Alcoholic HCl,† made by passing dry HCl gas into cooled absolute alcohol, acts readily on the carbides. The solution of the chlorides prepared by this method always contains more or less graphite, and is very difficult to filter because of its viscosity. By evaporation to dryness, the anhydrous chlorides are obtained, but care must be taken to prevent absorption of moisture, as the syrupy liquid is extremely hygroscopic. Several minor difficulties are encountered in this method, the principal ones being the solubility of acetylene in absolute alcohol (6 times as soluble as in water) and the lesser solubility of the chlorides in absolute alcohol (one-third as soluble as in water). Because of the above disadvantages, and the expense involved in the use of absolute alcohol and its subsequent loss on evaporation, this method was not suitable to large scale operations. The action of HCl gas and chlorine on the carbides gives chlorides which are highly contaminated with carbon and are therefore useless for electrolytic purposes, as carbon in a finely divided state with the chlorides will form carbides on electrolysis.

* Moissan (C. R. **124**, 1233) states that pure CeO_2 (free from iron and other earths) may be prepared from cerium carbide by fractional treatment of the carbides with dilute HNO_3. A modification of this method for the preparation of cerium dioxide free from the other rare-earths was made as follows. 100 grams of the mixed rare-earth carbides were ground to a fine powder and were treated with a small amount of dilute HNO_3. After the second treatment iron could not be detected in the residue by the sulphocyanide test. A larger amount of dilute HNO_3 was added and the solution after filtration was treated by Mosander's method. (Chlorine passed into the solution which had been made alkaline with KOH.) The precipitate after washing showed only a very faint absorption spectrum. For rapid preparation of cerium dioxide this method appears to be very good. The oxide prepared by this means is yellow when calcined at 100°, but turns darker when heated to 1000°.

† Meyer and Kess (Ber. **35**, 2622) have used alcoholic HCl for the preparation of cerium salts. They found that this solvent has no action on the oxides but reacts readily with the carbonates. They did not use the carbide.

In view of the fact that the non-aqueous methods were not suitable for the problem at hand, resort was again made to dehydration methods. It was found that, by treating the mixed oxides with concentrated HCl, evaporating to dryness, adding more HCl and repeating the process a number of times, a product was obtained corresponding to one of the lower hydrates of the chlorides. This white solid was very hygroscopic, and dissolved in water with avidity and evolution of considerable heat; when broken into lumps and calcined at a low temperature (not exceeding 125°) on an asbestos-covered hot plate further dehydration occurs without decomposition. The heating should be continued for a long time (about 12 hours), and the temperature must be kept low. If these directions are followed, a product corresponding approximately to the monohydrate is obtained. The great difficulty is to get rid of the last molecule of water, which is the most tightly bound of all, without decomposing the chlorides.

Matignon[40] and Bourion[41] have done a great deal of work on the preparation of anhydrous chlorides. They have found that certain chlorides of the metalloids, especially those of phosphorus and sulphur, act as catalysers in the dehydration of hydrated chlorides, and that in the presence of dry HCl gas, chlorine and sulphur monochlorides (S_2Cl_2) the chlorides of silicon, aluminum, thorium, neodymium, praseodymium, samarium and vanadium can be prepared in the anhydrous condition.

For the problem at hand this method had apparently many advantages and few disadvantages, chief of which was the very disagreeable nature of sulphur monochloride. It was resolved, therefore, to try out this method on the preparation of the anhydrous mixed chlorides. About 400 grams of the hydrated chlorides were placed in a porcelain tube furnace through which was passed a mixture of chlorine, S_2Cl_2, and HCl gases. The maximum temperature was about 300°, and the heating was continued for two and one-half hours. The product in the center of the tube was fritted together, and a sample for analysis was taken from this portion; 1.4 percent was insoluble, and the chlorine content of the soluble part was 42.4 percent—the percent of chlorine in $CeCl_3$ is 43.2 percent. The product melted satisfac-

torily, and the method seemed suitable, so that a plan was devised for its application on a larger scale.

A terra-cotta drain pipe, four inches (10 cm.) in diameter and four feet (1.2 m.) in length, was fitted at both ends with graphite caps, thru which passed short pieces of glass tubing, two at one end and one at the other. The furnace used was a circular-flame tube furnace in which the pipe could be heated gradually and uniformly, and the temperature was under positive control. The monohydrated chlorides, prepared by the method previously described, were dehydrated, about a kilogram at a time. The salt was spread evenly in the pipe, in a layer about ¾ inch (2 cm.) thick. The caps were then inserted and the ends heavily luted with a mixture of magnesia, asbestos fiber, cement, fire-clay and water, and allowed to dry, when a joint practically gas-tight was obtained. Dry HCl gas was passed over the chlorides, and as soon as the air was displaced the heating was commenced with a small smoky flame. After the tube was warm, chlorine and S_2Cl_2 vapors, as well as dry HCl, were passed thru the tube and the temperature was gradually raised to just below 400°. The dehydration is completed in about three hours if the temperature is correctly adjusted, but may take longer. The anhydrous chlorides are placed, while still warm, in dry bottles, and the corks paraffined in. Care must be taken to prevent the sewer-pipe from cracking when being heated, and this tendency forms one of the principal weaknesses of the process. A metallic container can not be used in the dehydration on account of the highly corrosive action of the vapors.

The sulphur monochloride seems to catalyse the dehydration process, and has the further function, together with the chlorine gas, of reconverting to chloride any oxide or oxychloride which may form. If the temperature is too high, oxide or oxychloride may form even in the presence of chlorine and sulphur monochloride. The dehydration may be carried out by HCl gas alone, and a scheme was devised for preparing pure anhydrous cerium chloride using this method. Since the process was the outcome of much work and of experience in the handling of the chlorides, it is described here in some detail.

The double cerium ammonium nitrate from the fractionating vats, where it has been purified and separated from the other

rare-earths by fractional crystallization, was used. The absorption spectrum of a concentrated solution of the nitrates showed only the faintest traces of the neodymium and praseodymium bands. The contents of a large bottle containing five kilograms of the double nitrates was divided into four portions and placed in fourteen-inch (35 cm.) porcelain evaporating dishes. The dishes were filled with concentrated HCl and placed on steam-heated sand-baths. Each dish was suitably protected from drippings from the hood by wooden covers raised a few inches above the tops of the dishes and supported on silica bricks. Fresh acid was added daily as required and the evaporation continued for about three weeks, at the end of which time the nitrates had

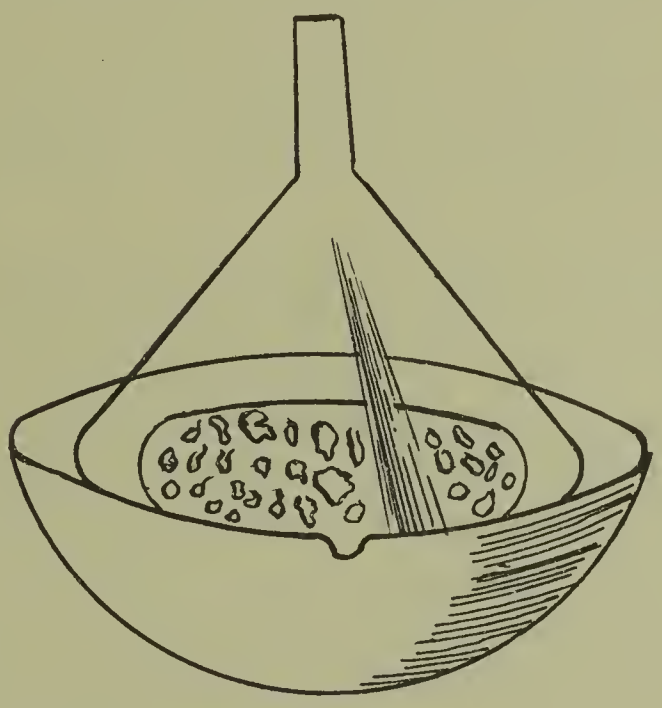

FIG. 1.

been completely converted to chlorides and all the ammonium salts had been expelled. The white solid was broken into lumps and heated on an asbestos-covered hot plate to convert it to the monohydrated chloride. The final dehydration was carried out as follows (see Fig. 1): About 400-500 grams were placed in a ten-inch porcelain evaporating dish. A short-stem funnel about nine inches in diameter was inverted and placed over the salt. A layer of asbestos fiber was packed tightly around the outside edge of the funnel and plaster of paris set over this. A glass tube passing thru the stem of the funnel and reaching to just above the layer of chloride conducted in the dry HCl gas, which was passed at a rapid rate over the heated chloride. The salt

was stirred around from time to time by means of a long, thick glass rod. The heating was done gradually, and the progress of the dehydration could be watched thru the glass funnel and its completion checked nicely. The anhydrous chloride was placed, while still warm, in dry glass vessels, and the corks paraffined in to exclude moisture from the extremely hygroscopic product.

The chloride prepared by this method melts to a clear, limpid liquid, and is suitable for electrolysis. The process meets practically all the requirements,—it gives a suitable product, it is rapid, inexpensive and simple, the progress and completion of the dehydration can be watched, overheating may be avoided, and large amounts of chloride prepared in the minimum amount of time. Chlorine and sulphur monochloride may be used in addition to the dry HCl gas, but this was not found necessary, as the water was driven off rapidly and completely without their presence.

It was observed that the anhydrous rare-earth chlorides may be partially converted into oxides or oxychlorides if they were carelessly heated in the presence of air. For this reason experiments were conducted on melting the chlorides in a non-oxidizing atmosphere in a Rose crucible. In the presence of carbon dioxide some oxychloride was formed, and in an atmosphere of carbon monoxide a crust of brownish oxide appeared.

The best method of melting the anhydrous chlorides without decomposition was found to be by heating a small portion in a covered crucible until a clear melt was obtained, and then introducing successive small portions of the chlorides to the melted portion. In this manner circulation of air is avoided, as the crucible is kept covered. A good way is to first melt sodium or potassium chlorides, or a mixture of these, and then to add small amounts of the rare-earth chlorides, keeping the crucible covered as much as possible. A small amount of oxide or oxychloride will "gum up" the entire melt, and for electrolysis this condition must be carefully guarded against.

Preparation of Anhydrous Fluorides.

Another salt required in large amounts was the anhydrous mixed fluorides, RF_3. This preparation involves some of the

difficulties encountered in the preparation of the anhydrous chlorides. The fluorides are more stable and less hygroscopic than the chlorides, but they are more corrosive in their action on apparatus. Porcelain vessels can not be used, and one is almost entirely limited to lead vessels in the preparation of these fluorides. However, lead linings are easily placed in evaporating dishes, especially in the large enameled iron ones, and five-gallon jars made of lead containing 10 percent antimony were found very convenient in this work.

A few methods are found in the literature for the preparation of anhydrous cerium fluoride, CeF_3, but not many. The fluorides of cerium, especially the hydrated ones, are insoluble in water and are gelatinous precipitates usually obtained by double decomposition.

The tetrafluoride $CeF_4.H_2O$ is found in nature as fluocerite, and when this mineral is heated it decomposes, forming the impure anhydrous fluoride CeF_3.[42] By precipitating cerous nitrate with hydrofluoric acid Jolin[43] obtained a gelatinous precipitate, which after drying over sulphuric acid corresponded to the formula $2CeF_3.H_2O$. When this substance was heated it decomposed, leaving a residue of oxide.

In the preparation of the anhydrous rare-earth fluorides both dry and wet methods were tried. Hydrofluoric acid gas was passed over a small amount of the pulverized carbides placed in an iron tube and heated to a temperature of about 200°. The hydrofluoric acid gas was prepared by heating CaF_2 with concentrated H_2SO_4 in a copper retort. Some fluoride was formed, but was contaminated with carbon and undecomposed carbide, and the method was regarded as unsuccessful.

The action of concentrated HF on the oxides was tried. A small amount of the mixed oxides was heated in a platinum dish with successive portions of concentrated HF. The action was vigorous at first, but soon stopped, and the residue was a mixture of oxide and fluoride.

As the anhydrous fluorides were required, a process in which no water was used obviated the subsequent removal of that water, and seemed to be at least worthy of trial. The chlorides were boiled with absolute alcohol until a concentrated solution was obtained, which was transferred to a lead tub placed in

hot brine, so that the precipitation might be done from a hot solution, and HF gas passed into the hot chlorides. The objections to this method are that the fluorides so prepared are contaminated with some chloride. Furthermore, the saturation of the hot alcohol with HF gas is a very slow process, and the fluorides are extremely difficult to filter on account of their gelatinous nature. If the precipitation is done in the cold, the fluorides come down in a slimy condition almost impossible to handle, hence the reason for precipitating from hot solution. While the precipitation may be done by the action of a concentrated solution of hydrofluoric acid on the aqueous solution of the chlorides, the fluorides settle far less readily in water than in alcohol. Consequently, on account of the above reasons, the next modification in the process was to dissolve the mixed oxides in concentrated HCl, concentrate the solution and add the requisite amount of concentrated HF to the hot solution. After allowing the precipitate to settle, the supernatant liquid was syphoned off, alcohol was added, the whole was stirred thoroughly, the precipitate allowed to settle, the liquid syphoned off and the treatment with successive portions of alcohol repeated several times. The fluorides were dried at a low temperature, and were pink in color. They melted at a fairly high temperature (about 900-1000°) to a clear liquid, and the process was satisfactory except for the time required for the precipitates to settle.

In order to flocculate the fluorides so that they could be more easily filtered, the precipitate of the fluorides was placed in a round-bottom Jena flask about half full of absolute alcohol, and heated in an autoclave at a temperature of 130° and a pressure of 90 pounds per square inch. The product so treated seemed to settle somewhat more rapidly, but filtered very slowly indeed.

After many trials, the most satisfactory preparation of the anhydrous fluorides was found to be best effected by the following method: About a kilogram of the mixed oxides was placed in a large porcelain evaporating dish and digested in a steam closet with concentrated HCl until a clear concentrated solution of the chlorides was obtained. The hot solution, containing only a slight excess of hydrochloric acid, was transferred to a five-gallon lead jar and concentrated HF added to complete precipitation. The fluorides were allowed to settle somewhat, as much of liquid

syphoned off as was possible, and washed twice with hot water and several times with 95 percent alcohol. Only a few minutes' time was allowed after each washing for the precipitate to settle. The fluorides were then placed in a lead-lined cage-centrifuge, and most of the remaining alcohol removed in this manner. Absolute alcohol was added and the solution evaporated to dryness without decanting. The absolute alcohol dehydrates the centrifuged fluorides very well. They were then dried at 100°, and the temperature raised to 200° at the end. About six kilograms of the fluorides were prepared in this manner. The process is a fairly satisfactory one, and as no filtering is done, the method is fairly rapid.

PREPARATION OF METALLIC CERIUM AND MISCHMETALL.

Thermal Reductions.

Thermal reductions were tried in the hope of producing rare-earth metal by this simple means. Previous work of this nature has already been referred to.[15-19] Magnesium and calcium shavings and aluminum powder were used as reducing agents on the mixed oxides. These experiments were conducted in electrically-baked magnesia linings placed in graphite containers. Carbon and silicon were also used to reduce the oxides, but external heat was applied to give the high temperature required. No metal was formed in these last instances but the carbides and silicides respectively. The carbide RC_2 is pyrophoric, but the silicide RSi_2 is not. Reductions using magnesium and calcium in excess gave alloys, many of which were pyrophoric.

Several trials to reduce the oxides with aluminum are here recorded:

$3R_3O_5 + 10Al = 5Al_2O_3 + 9R$

150 grams mixed oxides require 27 grams aluminum.

$3BaO_2 + 2Al = Al_2O_3 + 3BaO$

507 grams barium peroxide require 54 grams aluminum.

The following charge was weighed out, intimately mixed and fired by means of magnesium wire and thermit igniter.

Mixed oxides	150 grams
Aluminum powder	40 "
Barium peroxide	120 "

The reaction was vigorous, but not violent. A five-gram lump of metal and many small globules were obtained. The specific gravity of the metal was greater than that of aluminum.

In another experiment the following charge was used:

Mixed oxides	200 grams
Aluminum powder	61 "
Barium peroxide	200 "
Black thermit mixture	50 "

The black thermit was used to produce a higher temperature and also for the reason that the affinity of cerium for iron was thought to be greater than that of cerium for aluminum; 35 grams of hard-brittle alloy were obtained, but the content of rare-earth metals was low.

In the case of these thermal reductions, either an alloy, a compound or a lower oxide of the rare-earths was obtained. Owing to the high heat of formation of these oxides, the failure of alumino-thermics in the production of metal was only to be expected. The writer believes that a study of the equilibrium conditions of these reactions would form an interesting and valuable research and might result in the discovery of lower rare-earth oxides.

Earlier Work on the Electrolysis of the Anhydrous Chlorides.

The previous work on the electrolytic preparation of cerium has already been referred to. The method of Bunsen,[44] which the earlier investigators used, was first tried by the writer as follows: In a porcelain crucible, in which the mixed chlorides of sodium and potassium has been melted, small amounts of anhydrous cerium chloride were introduced by means of a small porcelain spatula. An annular piece of iron was used as anode, and the cathode consisted of a platinum wire, protected by a short piece of clay pipe stem. The temperature of the fused bath was lowered until a thin crust formed on the surface of the bath, when the electrolysis was commenced. Globules of cerium rose to the surface and burned with explosive force. The platinum cathode was badly attacked; in fact, part of it melted off, due to formation of an alloy with cerium. No metal was collected.

For a survey of Muthmann's work on the rare-earth metals the reader is referred to the original references.[23-24] The type of vessel used in the electrolysis of the chloride is shown in Fig. 2, and is now gnerally known as the Muthmann cell. It is a semi-water-jacketed vessel, constructed of copper, and may be used with or without the heating circuit. The principal objection which the writer has to the cell is its complicated nature.

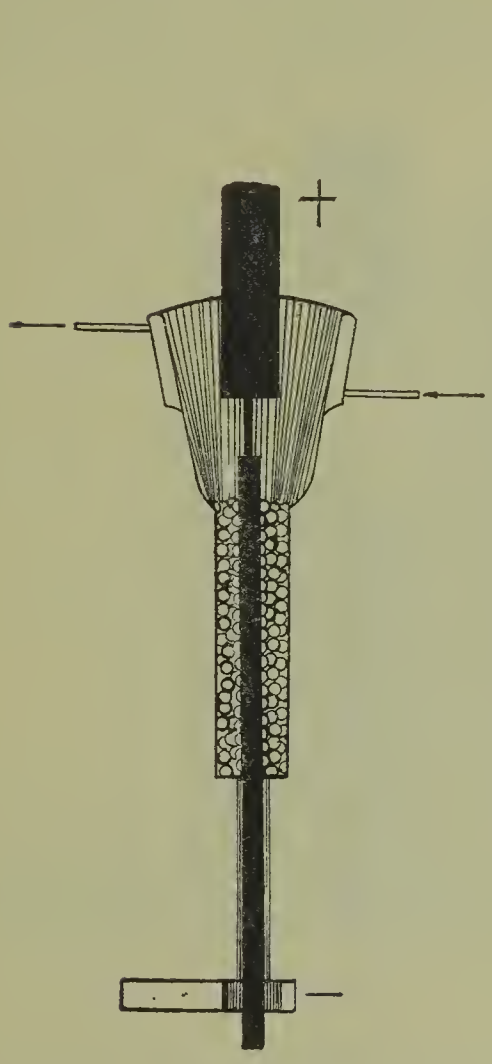

FIG. 2.

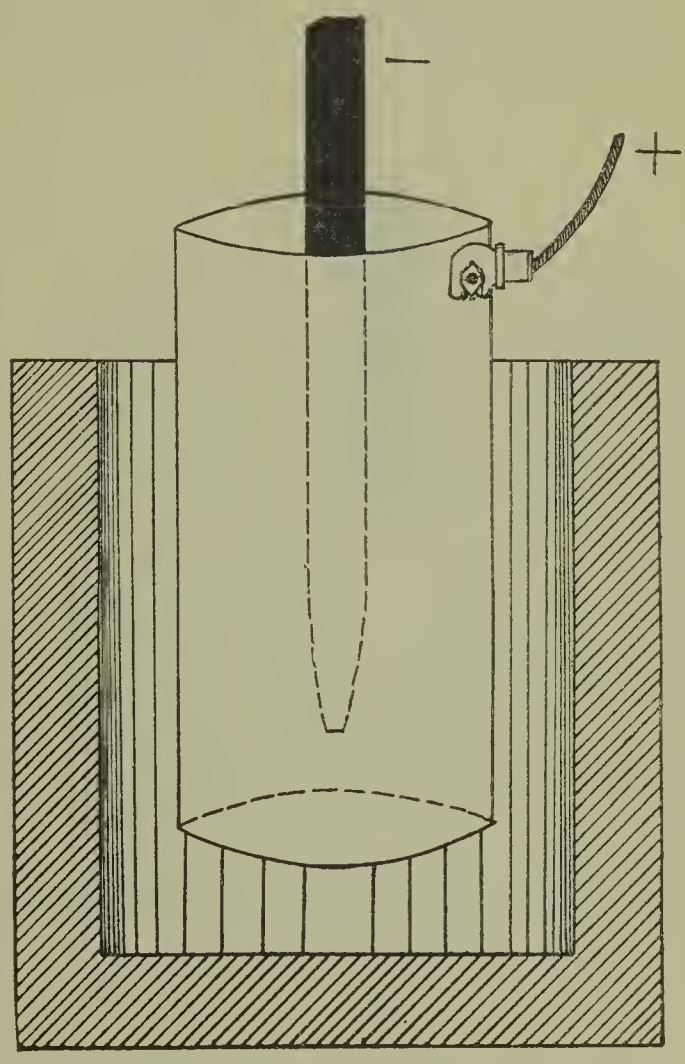

FIG. 3.

The cathode should be insulated carefully, and this is difficult, as the asbestos packing is attacked by cerium metal, and during the course of the electrolysis the insulation is apt to be destroyed and the walls of the vessel may act as cathode, with the subsequent contamination of cerium with copper. This is especially liable to occur if the temperature of the bath is too high. The heating circuit must also be insulated from the walls, and the carbon heating rods are liable to crack and break. Unless the

contents of the cell are poured or tapped at the completion of the electrolysis, the entire cell has to be dismantled after each run. When worked on a small scale, the manipulation of this electrolytic cell is a troublesome and difficult operation.

In the present research, numerous runs were made before more than traces of metal were obtained. It is believed by the writer that an account of failures is often fully as important as that of successes, as it is only by correction of faults that successful results are obtained. Therefore a brief account of the earlier

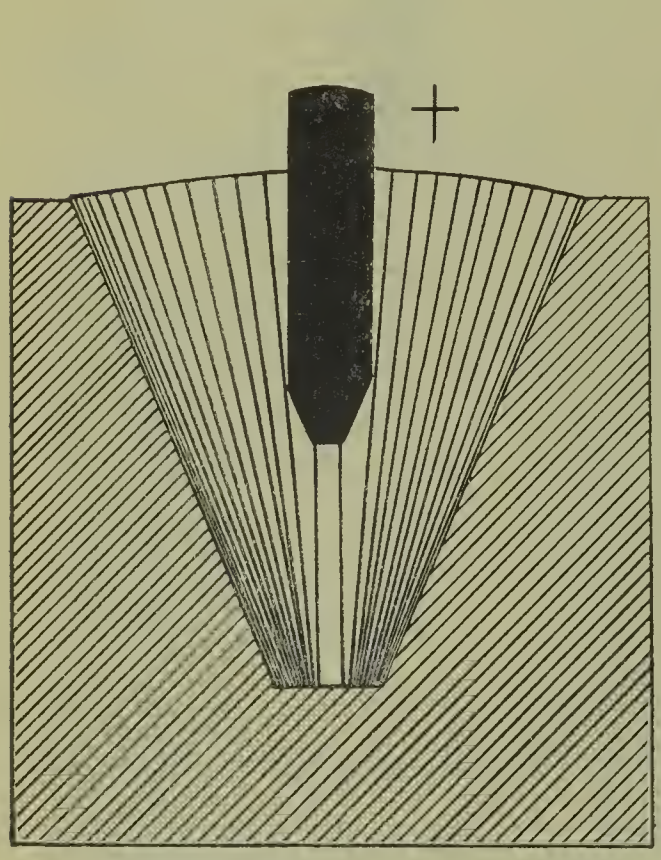

FIG. 4.

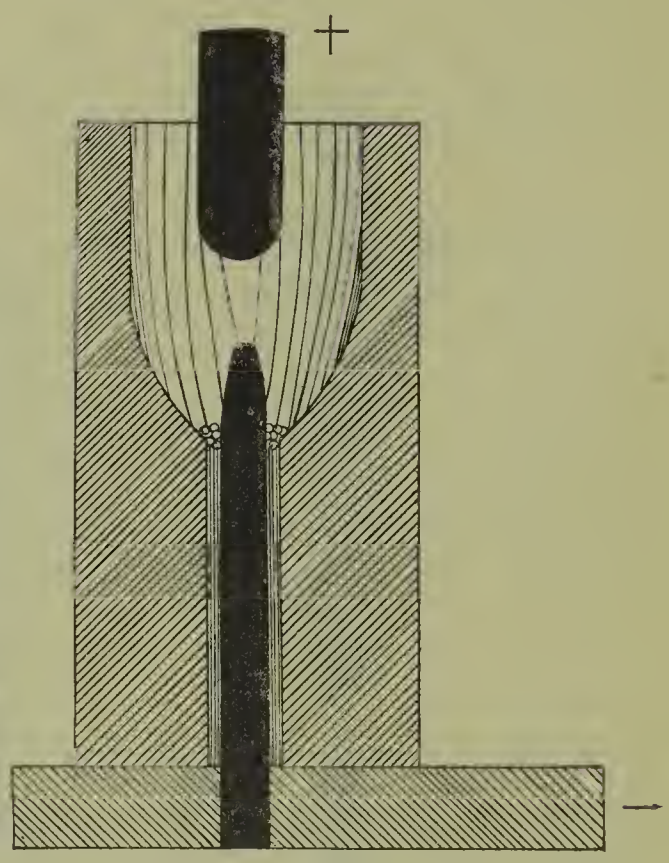

FIG. 5.

unsuccessful attempts to produce rare-earth metal are here given. It was only by a complete understanding of the reasons for failure that a successful solution of the problem was accomplished.

The different types of cells used where anhydrous mixed chlorides were employed as the electrolyte are shown in Figs. 3 to 5.* They were all constructed of the same material, graphite. The anodes were of graphite, but in the case of the insulated cathodes these were sometimes of graphite and sometimes of

* The crucible shown in Fig. 4 was split longitudinally into two sections, which were cemented together by graphite-molasses paste and clamped firmly together. This makes a very convenient form of electrolytic cell, as it can be easily dismantled, and also very quickly reassembled.

iron. In many cases where a graphite crucible was used as both containing vessel and cathode, the melting was done in a resistor furnace, and then the electrolysis was begun. In some instances the heating was done by alternating current, and could be maintained and regulated during the course of the electrolysis. The melting of the electrolyte was sometimes done by a small, thin carbon heating rod placed between electrodes, as shown in Fig. 2. An arc could not be used to melt the chlorides, as they are decomposed at that temperature.

The principal difficulties encountered in these experiments were the following:

1. The salt would not melt to a clear liquid.
2. The electrolyte would remain liquid for a time, but would eventually solidify.
3. The IR drop across the cell and the amperage would vary.
4. The anode would not functionate properly.
5. On completion of the electrolysis, no regulus of metal was obtained.
6. Minor difficulties, such as short-circuiting of insulated cathodes, leakages, fracture of electrodes, etc., liable to happen in any fused electrolysis, were encountered.

The reasons for the above difficulties were found by experience to be explained as follows:

1. If the chlorides did not melt to a clear liquid, then either the chlorides were not anhydrous or they were decomposed in the process of fusion. The proper manner of melting the chlorides has already been discussed in sufficient detail.

2. The "gumming-up" was caused either by the formation of a higher melting salt, as the oxide or carbide, or the temperature of the bath was too low. (See 4, below.)

3. As the generator voltage and the external resistance were constant, changes in the voltage across the cell and the amperage were caused by a variation of the internal resistance of the cell. This variation was caused by the following factors: Change in temperature of the bath; depletion in the electrolyte, or segregation, such as double layers, etc.; formation of intermediate products of varying resistance, such as sub-chlorides; differences in

thermal conductivity of the electrolyte at different stages of the electrolysis; etc. It was found that the potential difference and the amperage of any one cell can vary only between certain limits for successful electrolysis. The heating value of the current (C_2R) in cases where no external heat is applied must be such as to keep the temperature of the bath at a certain value. If the temperature of the electrolyte is too high, it is impossible to successfully electrolyse in the case of cerium chloride.

4. The function of the anode was (1) to cause the separation of chlorine and (2) to supply most of the heat necessary to keep the bath fused.

(1) In all these experiments the amperage was such that, if the cell was working properly, there was a copious evolution of chlorine gas. In many cases the voltage across the bath and the amperage would be approximately the proper values, and yet very little chlorine would be evolved. This may occur under several very different conditions. If the temperature of the bath is low, the semi-fused chloride is a very good conductor, and the entire bath may behave as a conductor of the first class. In this case practically no chlorine will be evolved. Again, if the temperature of the bath is too high, little chlorine is given off. This is due to the fact that at the cathode the chloride is partially reduced to subchloride, which diffuses to the anode and is there oxidized by the chlorine to chloride. There is a narrow limit of temperature where true electrolysis occurs, that is, where anhydrous $CeCl_3$ is decomposed by the current to metal and chlorine.

(2) In cases where the containing vessel acted as the cathode, anodic current density was much greater than the cathodic, consequently most of the heat was supplied to the cell by the anode. In the preliminary experiments the anodes were of graphite, but with the same diameter anode and the same current the heating is greater with a carbon than with a graphite anode. In some of the later runs carbon was used as the anode material for the reasons mentioned above. If the diameter of the anode is too great, a larger current is required to keep the bath molten. Besides, the anode is brought nearer the walls and most of the current may pass thru the upper part of the cell. This should be avoided, as the path of least resistance for the current should be from the anode to the bottom of the cell, as this insures good

circulation of the electrolyte and the deposition of the metal in a united form. If metal is deposited on the upper walls, some is apt to become oxidized by contact with the air, and this may result in the bath becoming viscous.

5. Many of the reasons for failure to obtain a regulus of metal have been given above. The metal may be deposited in a finely-divided, unfused condition, and may be disseminated throughout the body of the electrolyte. It may be totally or partially soluble in the electrolyte (for instance, Li in LiCl.), or the metal may react with the products at the anode (as chlorine) or the material of the cell (with copper forming an alloy, or with graphite forming a carbide).

Electrolysis of the Anhydrous Fluorides.

When it was found that the electrolysis of the chlorides was such a difficult process, it was thought that the electrolysis of some other salt might be more easily effected. Muthmann[24(c)] has used the solution of the oxides in the fused fluorides, similar to the process for the production of aluminum.

The anhydrous mixed fluorides melt at a temperature of a little over 900°, and dissolve the mixed oxides with avidity. The electrical conductivity of the molten fluorides is low, but increases rapidly with addition of oxides. The solution containing about 20 percent of oxides conducts very well.

The first cell tried with the fluorides contained a graphite cathode insulated from the graphite vessel by a porcelain ring. During the electrolysis the porcelain was badly attacked by the fused fluorides. Even a Dixon crucible was badly eroded, as the fluorides attacked the silicate binder. It was found that graphite was one of the few substances that would withstand the high temperature and the action of the fused fluorides. A vessel made of graphite plates, luted on the edges with graphite-molasses paste, clamped together with iron bands and previously baked, leaked during the electrolysis. Altho the vessel was imbedded in magnesia flour to prevent oxidation, the high temperature of the bath (well over 1000°) caused the luting to break down.

A summary of the results of the runs using fluoride-oxide electrolyte are here given.

1. 2,500 grams anhydrous mixed fluorides were melted in a

resistor furnace. The fused fluorides readily dissolved 450 grams of mixed oxides. The molten bath was electrolysed, using 450 amperes at a pressure of 14 volts. 10-15 grams of impure metal were obtained. The electrolysis was unsatisfactory, as the anode was not properly "wet" by the electrolyte. The active surface of the anode should have been much greater. A layer of carbide was found on the bottom and sides of the crucible.

2. A graphite box, inside dimensions 4½ x 5 x 6 inches (11 x 12 x 15 cm.), which was chiseled from a solid graphite block, was used as the electrolytic vessel. The anode was made as follows: A graphite block about 16 in. (40 cm.) long, 3 in. (8 cm.) wide and 2 in. (5 cm.) thick was cut at one end so as to present three blades, each ½ in. (1 cm.) thick and 3 in. (8 cm.) wide. This increased the active surface of the anode and resulted in its functioning properly. The fluorides were melted by a carbon heater placed between the electrodes. As soon as a small amount of the fluorides was melted, the heater was removed, the anode lowered and alternating current turned on. 1,000 amperes at 24 volts for about 25 minutes were required to melt 4,300 grams mixed fluorides. Mixed oxides were added gradually, and then direct current switched on. 650-750 amperes at a pressure of 10 volts were used. Oxide was added gradually during the course of the run, which was continued for about an hour, at the end of which time the solution solidified. 4,300 grams fluoride and 1,600 grams oxide were used in all. Small amounts of metal were scattered throughout the mass, but no regulus was obtained. At the bottom of the vessel was found a black layer of carbide. The melting point of the fluoride-oxide mixture was about 1400°.

The principal difficulties encountered in the electrolysis of the oxides dissolved in the fused fluorides were:

1. Oxides must be added to the fluoride to make a sufficiently conductive solution, and the melting-point of the bath is very high.

2. At the high temperature of the bath, rare-earth metal and graphite combine to form carbide.

Attempts were made to electrolyse a solution of the oxides dissolved in a mixture of fused potassium fluoride and rare-earth fluorides. Violent explosions ejected a large portion of the elec-

trolyte during the run, due perhaps to the deposition of alkali metal, which was immediately vaporized at the temperature of the bath.

One of the principal difficulties in all the previous electrolyses had been the formation of carbides, which made the bath viscous and unfit for electrolysis. It was thought that if a non-carbon cell was used, the formation of carbide would be less likely to occur, as the only carbon in the cell would be the anode. On account of their corrosive action and high melting point the fluorides could not well be used in other than carbon vessels. It was therefore decided to try the electrolysis of the mixed chlorides in a wrought-iron vessel.

Later Work on the Electrolysis of the Anhydrous Chlorides.

1. The electrolytic vessel was a thin-walled wrought-iron crucible about 3 in. (8 cm.) high and 2 in. (5 cm.) average diameter. The anhydrous mixed chlorides were fused by means of a thin carbon rod, heated electrically to incandescence, and electrolysed with a current of about 50 amperes for several hours. The electrolyte remained fluid during the entire course of the electrolysis. About 30 grams of semi-fused metal was obtained.

The iron crucible was not attacked. The success of this run led to experiments on the best conditions for electrolysis on a larger scale.

2. The electrolytic vessel was a large iron crucible 5½ in. (14 cm.) maximum inside diameter and 7 in. (18 cm.) high. The anode was a carbon rod 1½ in. (4 cm.) in diameter. A small amount of sodium chloride was fused, and the mixed chlorides were added in small amounts until a fused bath was obtained. The electrolysis was maintained with a current of 130 amperes for four hours at a pressure of 12-18 volts. Small amounts of anhydrous potassium fluoride (about 10 grams at a time) were added occasionally to dissolve what little oxide was formed and so keep the bath perfectly fluid. The amount of additional salts used (KF plus NaCl) in all was about 10 percent by weight of the rare-earth chlorides. A lump of partially fused metal was obtained, which when re-melted under NaCl weighed 120 grams. The number of ampere hours used was 520, and the

ampere efficiency was 13½ percent. The crucible was not attacked.

3. The object of this run was to try the value of an insulated cathode. Previous experience had taught that if the insulated cathode was iron and a fairly large current used (about 200

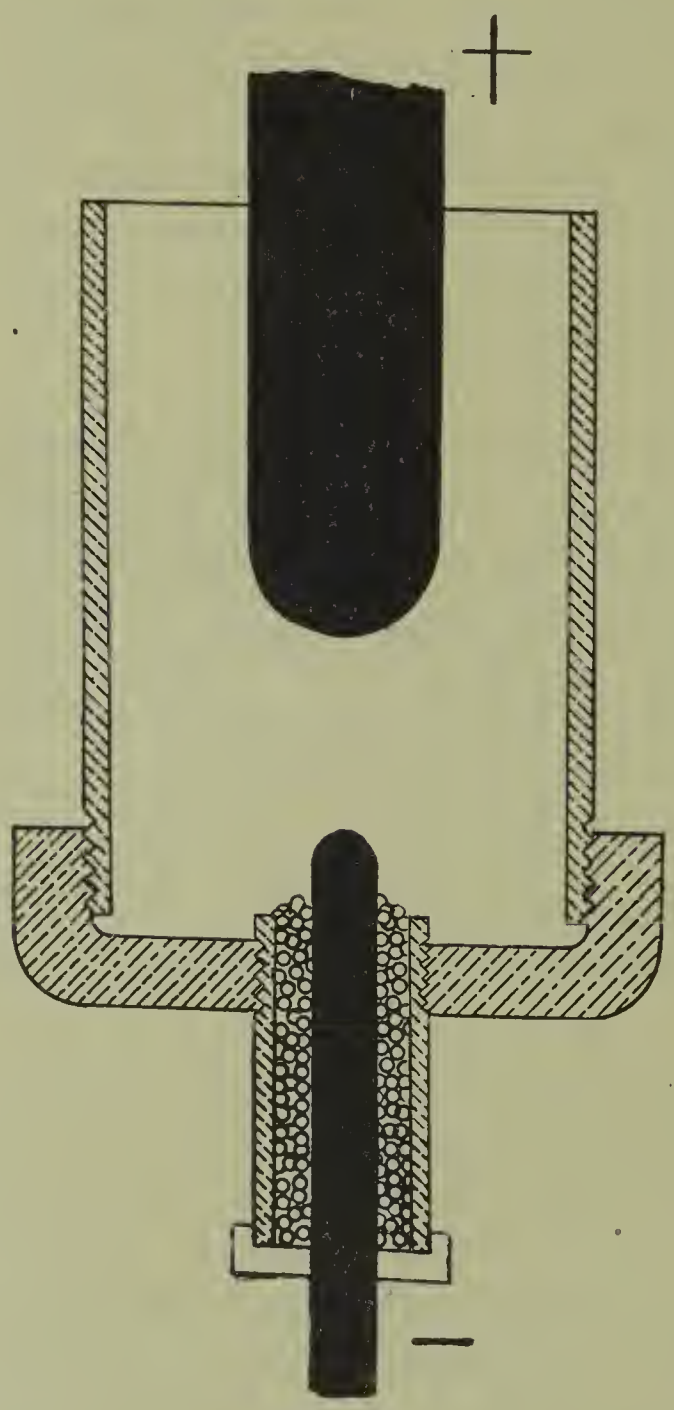

Fig. 6.

amperes) the iron cathode would become so hot that an alloy would be produced. Consequently iron was not used as the cathode in this electrolysis. The electrolytic vessel consisted of a 6-in. (15 cm.) length of a 3-in (8 cm.) iron pipe screwed into a reducing-cap carrying a short piece of 2-in. (5 cm.) pipe. The cathode consisted of a long rod of 1½-in. (4 cm.) graphite

insulated from the sides of the 2-in. (5 cm.) pipe by asbestos fibre and encased at the bottom in plaster of paris. A sketch of this cell is shown in Fig. 6. The anode was a graphite rod 1½ in. (4 cm.) in diameter. A current varying from 180 to 200 amperes at a pressure of 20 volts was used for nearly four hours. A regulus of metal weighing 135 grams was found at the bottom of the vessel. Below the metal was a black layer of carbide. The number of ampere hours was 712, and the ampere efficiency was 11 percent. The electrolyte consisted of the mixed chlorides, to which were added, during the course of the run, small amounts of anhydrous potassium fluoride, sodium chloride and barium chloride. The object of the last-named salt was to increase the resistance of the bath so that the electrolyte was kept well fused and metallic conduction of the bath prevented. It must be remembered that where a graphite anode was used, a higher voltage was required to keep the bath fused.

The results of the above three experiments showed: (1) That rare-earth metal could be produced by electrolysis of the chlorides in iron vessels; (2) that the use of an insulated cathode of graphite gave a lower current efficiency and resulted in some of the metal being converted to carbide; (3) that it was necessary to regulate the temperature of the bath so as to have the electrolyte well fused and yet well below the alloying temperature of iron and rare-earth metal; and (4) that control of the electrolysis could be maintained by judicious addition of small amounts of potassium fluoride, sodium chloride and barium chloride.

The above experiments in which the anhydrous mixed chlorides were used were so successful that the method of electrolysis was applied to pure anhydrous cerous chloride, and the runs are here described.

1. The electrolytic vessel was a thin-walled wrought-iron crucible, maximum diameter about 4 inches. The anode was a round graphite electrode 1 inch in diameter, and the iron crucible was made the cathode. A small amount of sodium-potassium chloride was first melted, and then cerous chloride added. During the course of the electrolysis, rather large amounts of the alkali chlorides were used. A current of 110 amperes was employed for two hours, at the end of which time the electrolyte solidified. Forty grams of fairly-well-fused metal was obtained.

The number of ampere hours was 220, and the ampere efficiency was 11 percent. Too much alkali chlorides was added during the electrolysis, and this fact, together with the high temperature of the bath,* accounts for the low efficiency.

2. A crucible similar to the one described in the above experiment was used both as the containing vessel and cathode. A corrugated graphite anode 5/8 in. (1.6 cm.) in diameter was used. 250-300 grams of anhydrous cerium chloride and about 10 percent sodium chloride were used. The electrolysis was continued

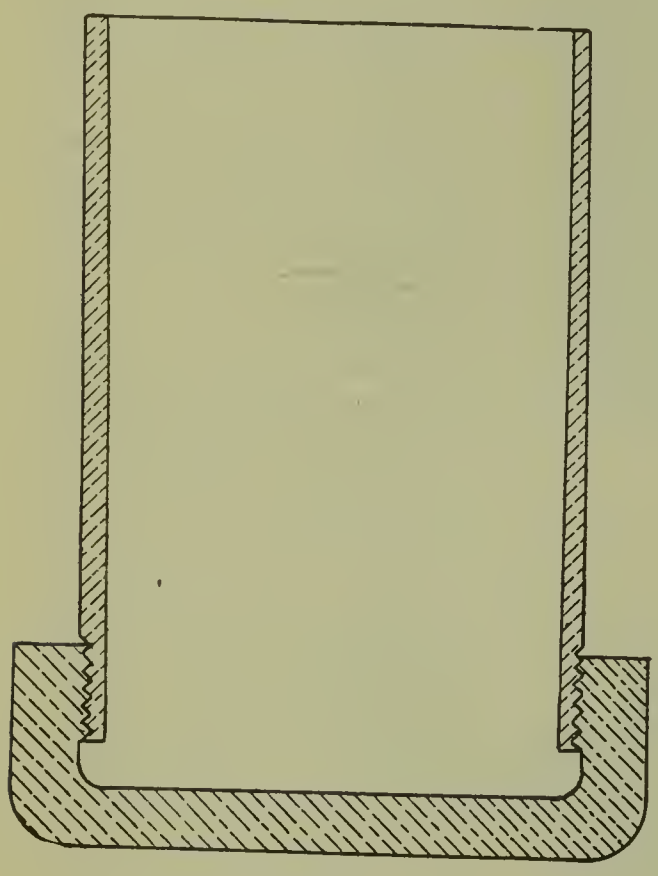

FIG. 7.

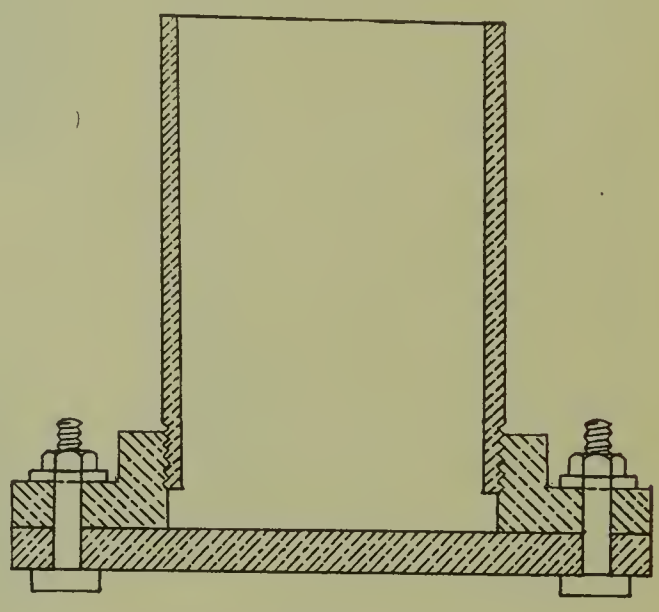

FIG. 8.

for 1½ hours, using 75 amperes at 15 volts. The bath was kept near its melting-point, so that there was no danger of a cerium-iron alloy forming. 65 grams of cerium metal was obtained. The number of ampere-hours was 112, and the ampere efficiency was 33 percent.

3. It was decided to try the electrolysis on a larger scale. When the mixed rare-earth chlorides were electrolysed in a large iron crucible, it was difficult to remove the solidified salt after

*In many electrolyses a high ampere efficiency is obtained only if the bath is kept near its melting point. As the temperature is raised the efficiency decreases. This is especially true in the case of fused lead chloride and fused caustic soda, where, if the temperature of the bath is high, no metal at all is obtained.

See these transactions, **19**, 167-168, 1911 (Discussion).

the run was completed. The chlorides, after cooling, form a hard, vitreous mass, upon which even a cold-chisel has little effect. The removal of slag and metal was always accomplished with difficulty and loss of some material. To obviate this difficulty a 7-in. (18 cm.) length of a 3-in. (8 cm.) iron pipe, screwed into a cap for a bottom, was used as the electrolysing vessel. (See Fig. 7.) Sodium chloride was melted first, and cerous chloride added until the fused mixture conducted well, when the carbon electrode, 1¼ in. (3 cm.) in diameter, was lowered until it was within an inch or so of the bottom; the voltage then sank to the proper value, 12 volts, and electrolysis began. The volume of the bath, of course, constantly decreases during the electrolysis, and fresh cerous chloride must be added from time to time, About every forty minutes the anode was raised until it just dipped below the surface of the bath. This increased the voltage across the bath and caused a rapid heating of the electrolyte. After a few minutes the anode was lowered until the voltage was properly adjusted, when electrolysis recommenced. The voltage varied from 12 to 14 volts, and the average current used was 200 amperes, for four hours. The electrolyte became slightly viscous toward the end of the run, but the electrolysis proceeded satisfactorily, as was indicated by the appearance of the bath, the voltage across the cell and the evolution of chlorine. Over a kilogram of cerous chloride was used. A well fused ingot of metal weighing 580 grams was obtained. The number of ampere hours was 800, and the ampere efficiency was 41½ percent. The cap could not be removed at the completion of the electrolysis, but the crucible was unattacked by the cerium. The small amount of iron introduced was caused by oxidation of the heated vessel near the top, the solution of this iron oxide in the electrolyte and its immediate reduction to metal when in contact with cerium.

4. In order to facilitate the removal of electrolyte and metal at the end of a run, the following vessel was devised: It consisted of a 3-in. (8 cm.) iron pipe screwed tightly into a 5-in. (13 cm.) iron flange, to which a bottom plate was attached by means of four large bolts and nuts. (See Fig. 8.) The electrolyte was cerous chloride containing a small percentage of sodium chloride, to which very small amounts of potassium fluoride and potassium

fluoride-barium chloride mixture were added when required (about every half hour or so). The function of these additional salts has already been explained in detail. The voltage was kept at its proper value, which should not be greater than 15 volts. The average current used was 200 amperes, for three hours. The electrolysis required attention about every twenty minutes; that is, the anode needed slight adjusting or the temperature had to be corrected. An ingot weighing 380 grams was obtained. The number of ampere hours was 600, and the ampere efficiency 36.5 percent.

The electrolyte remained liquid during the entire course of the electrolysis. About 1½ kilograms of cerous chloride was used. The cell held perfectly, and at the end of the run the electrolyte and metal were easily removed. This type of electrolytic vessel is highly recommended for similar work. Care should be taken that the flange and bottom plate are well faced and that the lap is wide enough (at least 1½ in. (4 cm.)).

ANALYSIS OF CERIUM AND "MISCHMETALL."

Two methods of analysis were used in this research for the determination of cerium.

1. *Gravimetric.* The cerium group was separated from all the other metals by precipitation of the oxalates in faintly acid chloride or nitrate solution, by means of boiling oxalic acid. Calcination of the oxalates at blast lamp temperature gave the oxides (in the case of cerium the dioxide CeO_2) which were weighed.

2. *Volumetric.* A modification of Browning's[45] method was used. It depended upon the oxidation of cerous salt to ceric, in alkaline solution, by means of potassium ferricyanide. Browning determined the excess of ferricyanide.

The modification was as follows. A slight excess of a dilute solution of potassium ferricyanide was added to the solution of cerous salt, and KOH added. This precipitation was usually done in a bottle, which was afterwards centrifuged. The supernatant liquid was decanted thru a small Gooch filter to guard against any loss of precipitate. The ceric hydroxide was washed two or three times with hot concentrated KOH to remove the ferro- and ferricyanides centrifuging after each washing. The ceric hydroxide was then treated with potassium iodide solution,

whereby the cerium is reduced to the cerous state and free iodine liberated, which latter is determined by titration with thiosulphate. By this method cerium may be determined in the presence of thorium and the other members of the cerium group. This method is accurate to one-half percent.

Grams cerium present	Grams cerium found	Error Grams	Error percent
0.22648	0.22697	0.00049	+0.2
0.22648	0.22657	0.00009	+0.04
0.22648	0.22697	0.00049	+0.2
0.22648	0.22752	0.00104	+0.5
0.22648 Ce 0.4 $Th(NO_3)_4$	0.22737	0.00089	+0.4
0.11328 Ce 0.10 $La_2(SO_4)_3$	0.11388	0.00060	+0.5

Analysis of a sample of "mischmetall" by the volumetric method showed a cerium content of 70.5 percent.

Analysis of metallic cerium prepared in Run 1, above, showed the following composition:

Percent Cerium	Percent Iron
97.5	1.2
97.8	
97.8	

The determination of cerium by the gravimetric method in the cerium metal prepared in Run 4, above, showed 97.8 percent cerium.

Analysis of cerium cast into rods in an iron mold was as follows:

Percent cerium	93.6
" iron	4.5
" insol residue (oxide)	0.53
" magnesia (from MgO lining)	0.4
" carbon	0.88
" chlorine	0.07
	99.98

PURIFICATION OF METALLIC CERIUM.

The chief impurities of cerium (amounting in the aggregate to about two percent) obtained by the method previously described consisted of iron, cerium oxide and cerium carbide, of which over one percent was iron.

Owing to the fact that metallic cerium is but slowly attacked by concentrated sulphuric acid, filings of the metal were boiled

with this acid. All of the iron was not dissolved, and the method is worthless for purification purposes.

The magnesium alloys of cerium, containing from 50 to 85 percent cerium, are brittle and may easily be pulverized to a fine powder. When digested with strong sulphuric acid for a few hours, the residue contains cerium, magnesium and iron. This method is also worthless.

The best method of purification consists in preparing the cerium amalgams. The amalgams are prepared by boiling mercury with cerium in a long iron pipe arranged with a condensing tube at the top. Solid amalgams are easily obtained by this method. The iron and impurities float to the top and may be skimmed off. The amalgams carefully prepared give only a very slight test for iron. The cerium and mercury may be separated by distilling the latter, but this must be done in a high vacuum to prevent oxidation of the cerium. A high temperature is required to drive off all the mercury. The amalgam should be placed in a magnesia vessel, and this in turn heated in an evacuated quartz vessel. The high temperature required would cause the collapse of an evacuated glass vessel.

PHYSICAL PROPERTIES OF METALLIC CERIUM.*

In the determinations of the physical properties, metal of approximately 98 percent of cerium was used, unless otherwise specified.

1. *Atomic Weight.* The atomic weight of cerium, determined from pure salts, is 140.25.

2. *Specific Gravity.* The specific gravity determinations were made in a 50 c.c. pyknometer of cylindrical form, with thermometer ground in central neck and with fused-in capillary tube at side. The liquids used were pure benzene and toluene, which have no chemical action on cerium. Several different specimens of metal were used for these determinations. The weight of metal taken varied from 3.4 to 11.2 grams. The average of determinations gave 6.92, at 25° C. Muthmann[56] found the specific gravity at 20° equal to 7.0424, and Hillebrand[57] gives a value of 6.728 for his remelted cerium.

*Some of the physical and chemical properties of cerium have been determined by Hillebrand, 21 and 55, and by Muthmann and his associates, 23-24.

3. Molecular State of Aggregation. No reference was found in the literature as to the crystallographic properties of cerium. Attempts were made by the writer to obtain crystals, but without success. The interior of many ingots of cerium were highly crystalline, especially in the center of blow holes. The best condition for crystal growth is slow cooling from the molten condition. In order to aid the formation of crystals, a small quantity of cerium was melted under lithium chloride. The crucible was kept just hot enough to prevent the lithium chloride from

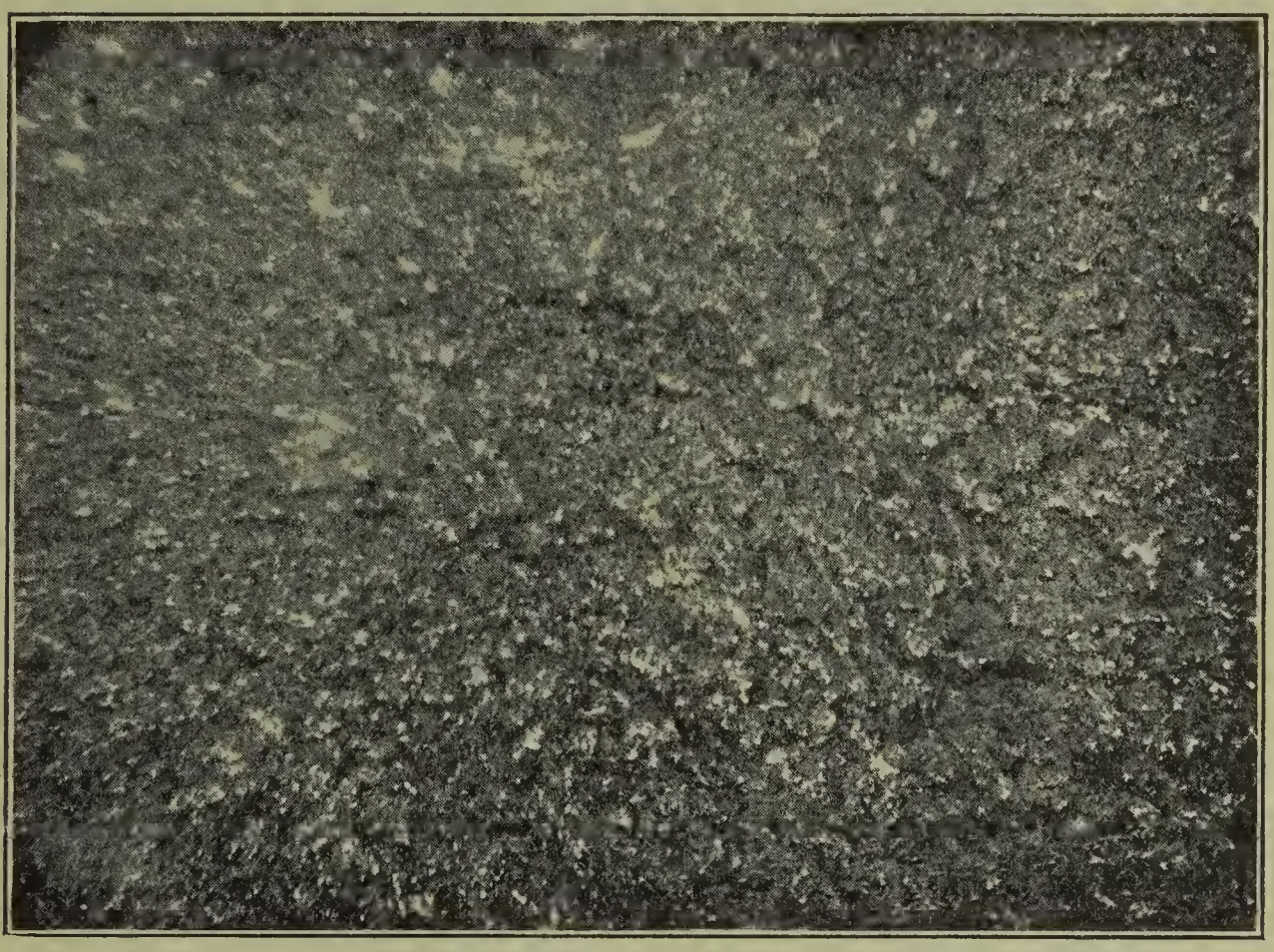

FIG. 9. × 70

solidifying, and was maintained at this temperature for several hours. The melting point of lithium chloride (about 550°) is somewhat below the melting point of cerium, and under these conditions the formation and growth of crystals would be expected. However, no definite crystals of cerium were obtained.

In order to examine the inner structure of cerium, photomicrographs of different specimens were made. The specimens were prepared by dry-polishing, and were etched with alcoholic HCl. An examination of these photomicrographs (see Fig. 9) shows homogeneity of structure.

4. Hardness. For the determination of hardness the scleroscope was used. The hardness of cerium varies, depending upon whether the surface of the metal is rolled, freshly cut, or old. The average value of the hardness for rolled surface was 25.9. The average for freshly cut surface was 9.5. (The reading 100 is the hardness of the standard steel test plate.)

5. Tenacity. Cerium is malleable and highly ductile. A strip of cerium was rolled to a thin sheet of a thickness of 0.015 mm. Cerium metal can easily be cut with a knife or scissors, and can be machined fairly well, although there is some tendency for the metal to buckle, as does lead.

6. Electrical Conductivity. In order to make these measurements, a test bar was necessary. A good method of obtaining a known length and cross-section of metal is to take a short piece of capillary tubing, apply suction at one end, insert the other in the molten metal and so fill the tube. On account of the corrosive action of cerium on glass and silica, it was impossible to use this method.

It was difficult to cast cerium into solid rods, owing to its rapid oxidation in air. The following mold was prepared, which was so designed that molten cerium could be quickly poured into it from the melting crucible and oxidation prevented: A wrought-iron pipe, ¼ in. (0.65 cm.) inside diameter and about 5 in. (13 cm.) long, was screwed into an iron base. A piece of cast iron was turned on the lathe to the shape of a small funnel, and was screwed into the wrought-iron pipe. The cerium was melted in a magnesia-lined graphite crucible under a covering of fused salt. The mold was heated to a dull redness, and the entire contents of the crucible poured suddenly into the mold. Very little oxidation or burning of the metal occurred. A rod of cerium 3 in. (7.5 cm.) long was obtained. This metal contained 93.6 percent cerium and 4.5 percent iron. Altho the inside of the pipe had been carefully cleaned, it was impossible to prevent contamination of the cast cerium with iron. As it was necessary to pre-heat the mold, a small amount of iron oxide necessarily formed, and, on contact with cerium, was reduced to metal and entered the rod as an impurity. The rod was turned down to size on the grinder.

The electrical conductivity measurements were made by the

voltmeter-ammeter method. The potential drop was taken over one inch of test bar, and all measurements were made at room temperature. The current was varied from 6 to 18 amperes. The specific resistance of the sample was found to be 71.6 microhms per centimeter cube.

7. Magnetic Properties. Stefan Meyer[46] has measured the magnetic susceptibility of many metals, among them that of cerium. He gives the atomic magnetic susceptibility of cerium ($K \times 10^{-6}$) equal to 34. He does not state the purity of the cerium used. The writer made no attempt to measure the magnetic susceptibility of cerium, but simply to determine if cerium is dia- or para-magnetic.

A small piece of cerium was suspended by a silk thread in a magnetic field of intensity about 5,000 lines per cubic centimeter. The metal so tested was paramagnetic, but not strongly so.

Cerium amalgam practically free from iron was sealed in a small glass tube and was tested in a magnetic field. It was weakly but definitely paramagnetic. From these determinations it was concluded that pure cerium is paramagnetic.

8. Melting Point. It was impossible to use the ordinary thermocouple method to determine the melting point, as no suitable protecting mantle could be found. Silica and porcelain are both attacked by cerium at its melting point. Magnesia, which is unattacked, is too porous a material for this purpose.

An attempt was made to obtain a cooling curve of cerium. About 60 grams of the metal were melted in a magnesia crucible in an electric resistor furnace and readings were taken with a small copper-constantan couple placed in a porcelain mantle. It was thought that altho the porcelain would be attacked somewhat, an approximate value for the melting point might be obtained. However, the cooling curve showed no constant temperature period and was worthless.

A small piece of cerium was attached to two long copper wires by means of small copper bolts and nuts, such as are used by watchmakers. Good electrical connection was obtained in this manner. The cerium was placed in a long Jena glass tube, sealed at one end and closed at the other by means of a rubber stopper, and so arranged that the tube could be evacuated. This was then placed in a tube furnace containing a calibrated thermo-

couple. A small amount of current was passed through the cerium, and when connection was severed, an electric gong sounded. This scheme of obtaining the melting point did not work, as the rubber stopper connection did not give a perfectly air-tight joint. The small amount of air in the tube combined with the cerium as the temperature increased. This caused a higher vacuum to be produced, with the result that more air entered the tube, and, upon subsequent repetition of this process, all the cerium was oxidized before the melting point was reached.

The approximate melting point was determined by heating a small quantity of cerium in a magnesia-lined graphite crucible and reading the temperature at which the metal softened by means of a thermocouple placed just above the metal. This crude method gave the approximate melting point as about 700°.

The real melting point of cerium was determined as follows: A small piece of cerium foil was fastened to two bent platinum wires by means of small copper bolts and nuts, and placed in a small glass bulb and the platinum wires sealed through the glass. The bulb was then evacuated by means of a mercury rotary pump, and sealed. A small incandescent lamp was connected in series with the cerium, which was heated in an electric muffle furnace. The temperature was read by a calibrated thermocouple. The melting point of the cerium was very definite, and occurred at a temperature of 635° C. The tube collapsed somewhat, but the method gave satisfactory results. The melting point obtained by the writer corresponds fairly closely with the value 623° C. given by Muthmann.[58] Hillebrand[57] says the melting point lies between those of antimony and silver.

9. Ultimate Strength. The same test bar was used for these determinations as in the electrical conductivity measurements. The ultimate strength was determined in an Olsen testing machine. The average diameter of the test bars was 0.212 in. (0.55 cm.). There was no elongation with constant load at 350 pounds (159 kg.). The test bar broke with a snap (like cast iron) at 495 pounds (225 kg.). The ultimate strength was equal to 12,900 pounds per square in. (9 kg. per sq. mm.).

10. Specific Heat. The specific heat was determined in a Joly differential steam calorimeter. The principal difficulty encountered was to get a suitable method of protecting the cerium

from attack by the steam. The cerium was sealed in a glass tube, but this method was unsatisfactory, owing to the time necessary for exchange of heat due to the small conductivity of the glass. Another method employed for protecting the cerium was to coat it with a mixture of amyl acetate in collodion. However, this coating was attacked by the steam and was liable to peel. The metal was also placed in a small nickel box, but was attacked by the steam. The best method of protecting cerium, at the same time allowing for rapid heat interchange, was to wrap the cerium tightly in several thicknesses of tin foil. Blanks were run on copper and tin foil to check the accuracy of this calorimetric method. The values for the specific heat of copper obtained with this steam calorimeter was equal to 0.0925. The specific heat of cerium (20-100°) was measured and found equal to 0.0524. Mendeleef[47] gives the specific heat of cerium equal to 0.05. Dr. W. F. Hillebrand[60] determined the specific heat of cerium by means of Bunsen's ice calorimeter. He gives corrected value of 0.04479 for pure cerium, and used only about 2 grams of metal in a determination. The writer used more than 70 grams of cerium in a determination, and obtained a corrected value of the specific heat of pure cerium equal to 0.05112.

11. Heat Conductivity. No measurements were made on the heat conductivity, and no references were found in the literature relative to this property. The heat conductivity seems to be fairly high, and, if so, cerium is probably one of the exceptional metals which has a low electrical conductivity and high thermal conductivity.

12. Latent Heat of Fusion. From observations on melting cerium, the latent heat of fusion seems to be fairly high. It has not been determined to my knowledge.

13. Heat of Oxidation. The heat of oxidation was determined in a Mahler bomb calorimeter. Cerium must not be burned in contact with platinum, as it will alloy with the latter. The cerium filings were placed in a small magnesia crucible in the calorimeter. From one to two grams of cerium filings were used for each determination, under an oxygen pressure of about fourteen atmospheres. The average value of the heat of oxidation was 1,740 calories per gram, or 60,900 calories per gram equivalent (CeO_2 = 243,600). These values are probably

correct within 4 or 5 percent. It is difficult to obtain an accurate value for the heat of oxidation where the products of combustion are non-volatile. The determinations in the case of cerium were unusually difficult to make, for the following reasons: The metal should be in a finely-divided form, but preferably in the shape of filings. When cerium is filed, care must be taken to prevent oxidation, owing to the low kindling point. A small amount of oxide forms in any case, and therefore makes the sample of metal taken non-uniform. If the filings are too coarse, oxide will fuse on the surface, and the metal in the interior will be unoxidized. Many of the difficulties encountered in the determination of the heat of oxidation of silicon[48] were

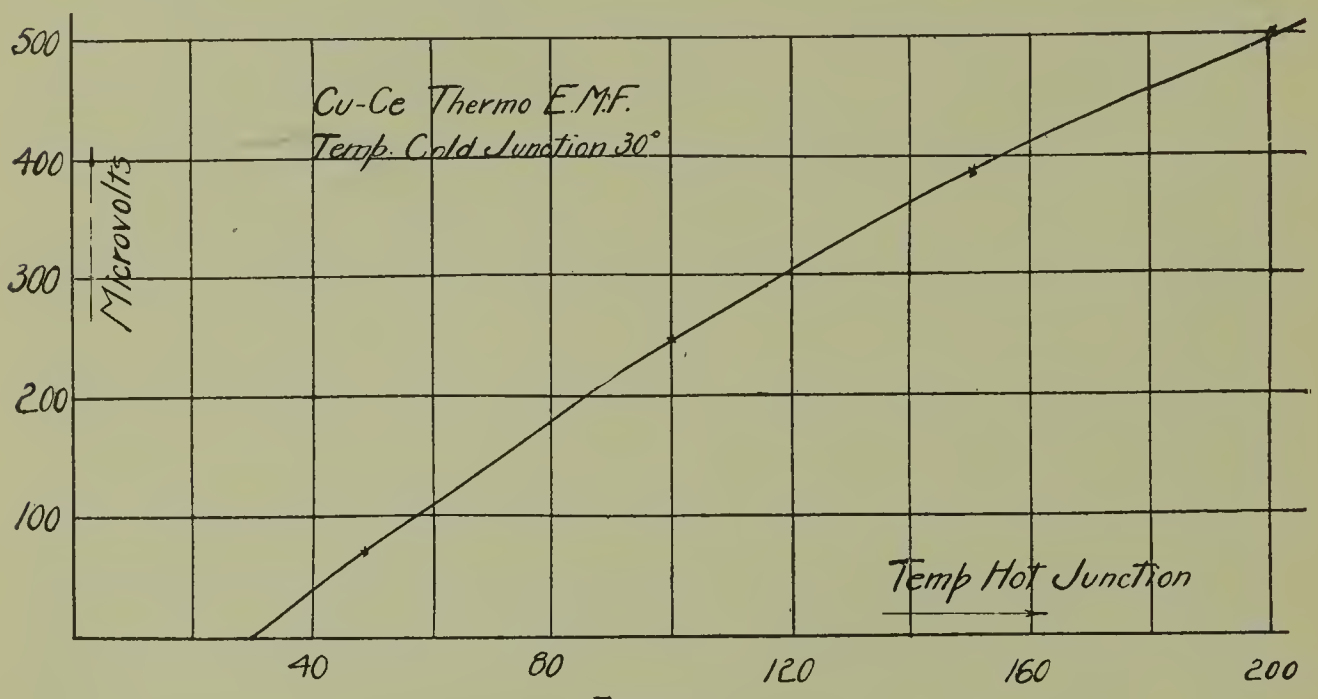

FIG. 10.

met with here. Muthmann[59] gives the heat of oxidation of cerium equal to 1603 cal. per gram.

14. Single Potential. Attempts were made to measure the single potential of cerium in the alcoholic solution of cerium chloride. No reliable measurement could be obtained, owing to the rapid formation of a film on the polished surface of the metal as soon as current flowed. The potential rapidly decreased as soon as the film appeared, which is almost instantaneous with the passage of current.

Measurements on the decomposition potential of the anhydrous mixed chlorides in absolute alcohol were made, using platinum electrodes. Gas was evolved at an applied electromotive force of 3.5 volts.

As the single potential of cerium could not be measured successfully, its value was calculated from the thermal data taken from Matignon's article on the anhydrous mixed chlorides:

Heat of formation of $CeCl_3$ (per 3Cl) = 270 calories
Heat of solution of $CeCl_3$ = 33 calories

The calculated decomposition potential of normal cerium chloride in aqueous solution is equal to 4.3 volts. Therefore the single potential of cerium in normal solution of its chloride measured against the normal calomel electrode is equal to —3.16 volts.

15. Thermo-electromotive Force. Two small glass cells, protected by asbestos and fitted with wooden covers, were used. Standard thermometers with small stirrers attached were inserted through the cover. Cottonseed oil was used for the bath, and one of the cells was heated internally by means of several coils of resistance wire. The electromotive force was determined by the potentiometer method with a special piece of apparatus specially prepared for this kind of work. The thermo-electromotive force of cerium against copper is given in the table below and in the curve shown in Fig. 10. The current flowed at the hot junction from copper to cerium. The measurements are given in the following table:

Hot Junction	Cold Junction	Electromotive Force
49.2 C	29.2 C	0.000075 Volt
100.	25.	0.000770 "
100.	Red. to 30.	0.000250 "
151.	26.9	0.000399 "
151.	Red. to 30.	0.000387 "
200.5	29.7	0.000504 "
200.5	Red. to 30.	0.000502 "

16. Optical Properties. Cerium alone could not be used, owing to the fact that a thin film of oxide forms on the polished surface. Different alloys of cerium were tried, and the best ones for this purpose were found to be the magnesium-cerium alloys. The preparation of samples had to be done very carefully, as fine emery and rouge entered the soft surface of the cerium. Dry polishing with a very fine file (the finest obtainable) was the most satisfactory way of preparing the sample. For the

methods of measurement of the optical properties the reader is referred to the article on the subject by Ingersoll and Littleton[49] on "A New Method of Determining the Optical Constants of Metals." The constants determined on the magnesium-cerium alloys are given in the table below:

Per cent Cerium	n	k
83	1.60	1.74
61	1.43	2.30
26	0.73	4.79
Pure Mg	0.32	12.0

CHEMICAL PROPERTIES OF METALLIC CERIUM.

Metal of approximately 98 percent cerium content was used in these determinations.

Cerium is very slightly attacked by cold water, but in boiling water a slow evolution of hydrogen gas occurs and the metal is tarnished black. At room temperature the following solvents have no action on cerium: Ethyl alcohol, amyl alcohol, chloroform, carbon tetrachloride, concentrated sulphuric acid, concentrated ammonium hydroxide, concentrated sodium hydroxide. Ethyl ether has a very slight action on the metal, as have also 3 percent and 30 percent hydrogen peroxide at room temperature. The action of dilute sulphuric acid, concentrated and dilute hydrochloric acid, concentrated and dilute nitric acid, in the cold, is moderately vigorous. Ammonium chloride and potassium chloride, at room temperature, have moderate action on the metal. This may be explained by the fact that in water a small amount of cerium hydroxide forms which is soluble in potassium chloride and ammonium chloride, thus causing fresh surface of the metal to be exposed and resulting in moderate attack of the metal by the solvent. At boiling temperature the following solvents did not attack the metal. Chloroform, carbon tetrachloride, concentrated sulphuric acid, concentrated ammonium hydroxide. The metal was only slightly attacked by the following solvents at boiling temperature: Ethyl alcohol, amyl alcohol, ethyl ether and concentrated sodium hydroxide. Dilute nitric acid, ammonium chloride, potassium chloride and 3 percent hydrogen peroxide at boiling temperatures gave moderate action. Dilute sulphuric acid, concentrated and dilute hydrochloric acid, concentrated nitric

acid and 30 percent hydrogen peroxide gave vigorous evolution of gas at boiling temperature.

The action of various gases on cerium was studied. A small amount of cerium filings was placed in a porcelain boat in a hard glass tube. The air was displaced by the gas under test, and the tube heated gently by means of gas burners. The cerium filings heated in chlorine emitted a very bright light at a temperature of 210° to 215° C. The salt formed was tested and found to be anhydrous cerous chloride.

In bromine, cerium burned at a temperature of 215° to 220° C. with emission of less light than in the case of chlorine. The salt formed in solution in water, and was identified as the bromide.

In iodine vapors no light was emitted, altho the tube was heated to a temperature of 300° C. On examination of the contents of the tube it was found that some iodide had formed.

Cerium burned with luminescence when heated in air to a temperature of 160° C. If a lump of cerium is sealed in a glass bottle and kept warm for some time, a black powder is seen to form upon the surface of the cerium, and when the bottle is opened this powder ignites at room temperature. This is probably due to the formation of a highly pyrophoric suboxide of cerium.

When cerium filings are heated to 345° in hydrogen gas, the hydride forms without emission of light. The hydride has been described and studied by Muthmann.[50]

Cerium filings were heated in nitrogen to a temperature of 1000° C. No luminescence was observed, but some nitride was formed. This nitride is a black powder, and has been studied and described by Matignon.[51]

When the nitride is heated in potassium hydroxide solution, evolution of ammonia gas occurs. When exposed to air the nitride gradually changes over to a brown oxide.

Cerium filings were heated in the vapors of sulphur. When the black substance which is formed is treated with dilute sulphuric acid, sulphuretted hydrogen is evolved. Cerium sulphide has been studied by various investigators.[52]

Cerium filings were heated to a temperature of 500° C. in an

atmosphere of carbon monoxide. There was no visible reaction, but part of the filings appeared oxidized.

ALLOYS.

A number of different alloys of cerium were studied, and are described below. In most cases the alloys were high in cerium, usually containing about 70 percent cerium. The metals were weighed out in the proper proportions, using from five to ten grams of cerium. The cerium was first melted in a small porcelain crucible under a layer of sodium chloride, and the other metal added in small pieces. The porcelain crucible was, of course, attacked by the cerium, but it was found that a thin layer of silicide of cerium forms, and this protects the crucible from further attack. The metal can in most cases be removed without breaking the crucible if care is exercised, and therefore one crucible serves for several melts. The silicide does not appear as a contaminant in the alloy. Porcelain crucibles were used except in cases where especially pure metal was desired, when magnesia crucibles were employed.

The work on the alloys of cerium is presented below in an epitomized form. The time at the writer's disposal was such that a complete study of each alloy could not be made. However, specimens of all the alloys described have been preserved and will be used for future references.

Muthmann and Beck[24b] have described some of the alloys of cerium with zinc, aluminum, magnesium and mercury.

Silver. Silver alloys with cerium quietly, and the thermal change appears to be small. The alloy has a silvery-white luster, and is hard and brittle. From all appearances, a compound is probably formed between cerium and silver.

Gold. When gold was added to molten cerium there was a slight flash from the crucible, but the reaction was not violent. The alloy was fairly soft, and had a reddish appearance. It disintegrated somewhat on ageing, forming a purplish-black powder.

Platinum. The reaction between cerium and platinum appeared to be endothermic. The alloy was fairly hard and was pyrophoric. It disintegrated to a slight extent when aged.

Copper. The cerium-copper alloy was hard and brittle, altho the two component metals were soft and malleable. A compound is probably formed. The alloy disintegrated to a powder.

Tin. This alloy was formed with the evolution of much heat. When first made, the alloy was pyrophoric, but on standing for a few months a powder formed, showing that the alloy disintegrated slowly when exposed to the air.

Antimony. There was considerable evolution of heat on the formation of this alloy. The alloy was very soft and was non-pyrophoric. It did not oxidize or disintegrate on ageing.

Arsenic. The reaction appeared to be exothermic. The alloy was fairly soft and somewhat pyrophoric. It did not decompose on being kept.

Carbon. Carbon and cerium combine directly when heated together. The carbide has been referred to and described earlier in this article.

Silicon. Cerium and silicon form the silicide $CeSi_2$. This compound may be formed by the reduction of the oxide of cerium. Large amounts of the mixed silicides have been prepared by the writer. The following mixture is recommended for their preparation:

M_3O_5	1,000	grams.
Powdered graphite ...	200	"
Powdered silicon	450	"

The silicide of cerium is somewhat brittle, and may easily be pulverized to a fine powder. It is a splendid reducing agent. When the silicide is added to cerium so that the silicon content is about 15 percent, a good pyrophoric alloy is obtained.

Sulphur. The preparation of the sulphide has been referred to earlier in this article.

Selenium. Some reference is found in the literature to the selenide of cerium. Mosander reduced the selenite in a stream of hydrogen and obtained a brown substance of a disagreeable odor. Moissan has shown that the selenides may be obtained by the action of selenium fumes on cerium carbide.

Selenium was added to molten cerium. The reaction was very

vigorous, and was accompanied with the evolution of reddish fumes. The melt consisted of two portions, a powder and a metallic alloy. The powder was impure cerium selenide, and the alloy, which was pyrophoric, apparently consisted of the selenide dissolved in excess cerium.

Tellurium. No reference was found in the literature to the telluride of cerium. Tellurium combined vigorously with molten cerium, giving a brownish pulverent mass, which was probably

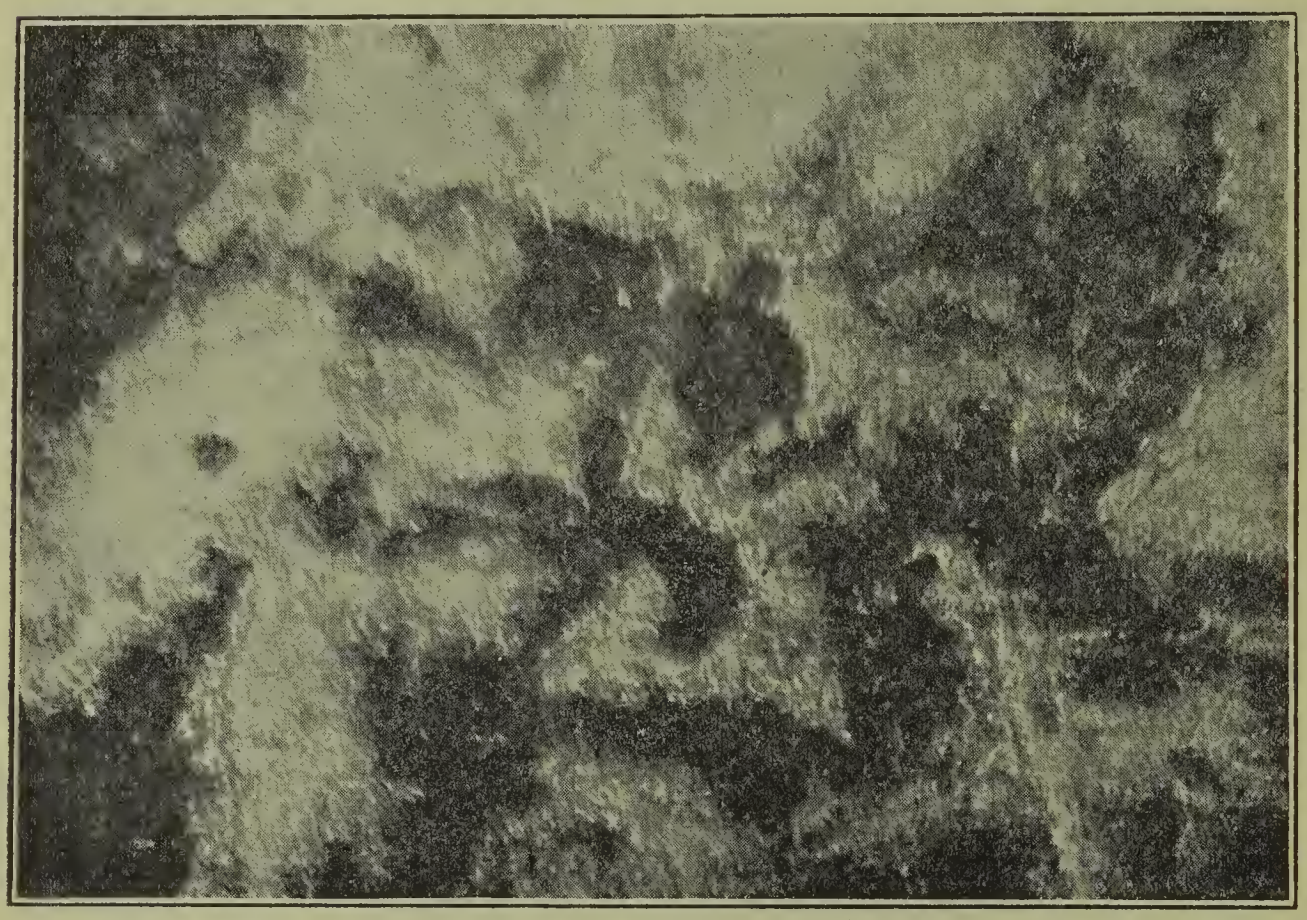

FIG. 11. × 200

the impure telluride. When treated with dilute acids, the well-known odor of hydrogen telluride was detected.

Lead. When lead was added to molten cerium, a violent reaction occurred, causing a portion of the contents to be ejected from the crucible. The alloy was very soft, and emitted sparks of a reddish color when scratched with a file. The alloy disintegrated slightly on ageing.

Calcium. Calcium alloyed with cerium quietly, without evolution of much heat. The alloy was white and was harder than either constituent. When scratched with a file it emitted a

cluster of bright sparks. It is stable in air, oxidizing very slowly. The alloy did not disintegrate.

Sodium. Sodium alloyed with cerium quietly. The alloy was hard and slightly pyrophoric. It oxidized somewhat when exposed to air.

Aluminum. Aluminum alloyed with cerium fairly quietly. The alloy was very hard and brittle. It disintegrated without oxidation. A compound of aluminum and cerium was probably formed. This alloy was not pyrophoric. Muthmann[61] describes a compound $CeAl_4$.

Zinc. The combination of cerium and zinc was accompanied by a vigorous, in fact, almost explosive, reaction. There was a large amount of heat evolved. The alloy was hard, brittle and pyrophoric, and remained unoxidized when exposed to atmospheric conditions.

Cadmium. Cadmium and cerium combined vigorously with considerable heat evolution. The alloy was hard, brittle and pyrophoric. On exposure to air a film of oxide formed slowly, but the alloy did not disintegrate.

Chromium. The alloy was white in color, hard and brittle. It was somewhat pyrophoric. It remained unchanged in the air.

Manganese. Manganese combined quietly with cerium, forming a fairly hard, pyrophoric alloy, stable in air.

Iron. The iron-cerium alloys are very interesting, as they were the first pyrophoric alloys known, and were discovered by Dr. Auer von Welsbach. The alloys of about 70 percent cerium content are fairly hard and somewhat brittle. The microstructure of one of these alloys is shown in Fig. 11. It is seen that the structure is heterogenous, probably consisting in part of a cerium-iron compound. These alloys have been the subject of some discussion in the literature,[53] as have the reasons for the pyrophoric properties.[54] The writer believes that the question of the pyrophority depends upon the following factors: Cerium alone is soft and malleable, and when scratched with a file, small particles are not broken off. However, when the alloy is hard and brittle, small particles are easily detached from the mass, and the friction is sufficient to raise the temperature of these small particles to the incandescent point of cerium (160°). As

metallic compounds (such as Cu_3Sn) are as a rule hard and brittle, the addition of such compounds to cerium usually assures a pyrophoric alloy. The alloy should be high in cerium, so that ignition occurs at a low temperature, and should contain excess of cerium above that required for the formation of a compound (such as $CeSi_2$) to act as a binder and so prevent the disintegration of the alloy, as in the case of the cerium-aluminum alloy containing about 60 percent cerium. The kindling point of the alloy may be lower than that of cerium.

Nickel. The nickel-cerium alloys made were similar to the iron alloys, but were somewhat softer. The ones high in cerium were very pyrophoric.

Tungsten. Powdered tungsten equivalent in weight to 15 percent was added to molten cerium. The tungsten dissolved fairly quickly and the reaction was quiet. The alloy was hard, brittle and pyrophoric.

Mercury. Cerium forms amalgams fairly easily. Their preparation has already been described under the methods of purification. The amalgams containing only 1 or 2 percent of cerium are liquid, but the ones of higher percentage are solid. These amalgams oxidize easily in the air, and those containing 8 or 10 percent cerium take fire in the air. Cerium will dissolve in mercury to the extent of about 15 percent.

Magnesium. The whole series of these alloys were prepared, and have been discussed under optical properties. The alloy containing about 83 percent cerium is highly pyrophoric. Most of these alloys are brittle and can be easily pulverized. Excellent flashlight powders can be prepared from the ones of higher cerium content. These alloys also form splendid reducing agents, as the combination of cerium and magnesium is an endothermic reaction, and when the alloy is oxidized, more heat is emitted than from an equivalent mechanical mixture of the two constituents. The fact that the alloys of from 60 to 75 percent cerium content may be easily pulverized in a mortar to a fineness of 200 mesh should render these alloys valuable for thermal reductions. A small amount of metallic vanadium was prepared by reducing V_2O_5 by this method. The alloys of from 75 to 85 percent cerium content can be pulverized, but they are so highly pyrophoric that it is difficult to prevent igniting them.

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58. Muthmann & Weiss, Lieb. Annalen, **331**, 36.
59. *Ibid.*, p 41.
60. W. F. Hillebrand, Pogg. Annalen, **158**, 71.
61. Muthmann & Beck, Lieb. Annalen, **331**, 47.

DISCUSSION.

PROF. C. L. PARSONS: I cannot help rising to say a few words in praise of the work Dr. Hirsch has accomplished. Anyone who has attempted to do anything with the old Muthmann method for the obtaining of cerium knows something of the difficulties which have to be overcome to get even a very small yield of the metal; and it is certainly a very decided increase in our knowledge to understand that, with a common piece of apparatus which anyone can make readily in his own laboratory from wrought iron pipe, we can get cerium in some quantity. Also, I want to say that I hope from this paper interest will be engendered so that we may find some uses for this metal, because cerium is not anywhere near so rare a metal as many people are inclined to believe. Certainly, cerium salts can be obtained in very large quantities, and there will be no difficulty whatever in making a great many compounds of cerium, especially as cerium is now probably the most easily separated of any of the rare earths. Taking the crude material as it comes from the Welsbach Company, cerium can be separated practically in a single operation over 99 percent pure, and accordingly we may hope to see cerium metal, if uses can be found, presented at a very low price. If you take a 30 percent solution of ceric bromate, the rest being water, and simply roll up a piece of filter paper so that it looks like a firecracker, and dip that into the ceric bromate, and hold it out, it goes off like a firecracker in about half a minute. I was in my laboratory one day when Professor James was working with some of that ceric bromate which he had been making an

effort to crystallize. He had it in his hand after partial evaporation in a vacuum, and while stirring it the rod went through the bottom of the beaker, and the contents went down on his shirt sleeve. There was a series of pyrotechnics that I can assure Dr. Hirsch beat that which they had with the specimen of which he spoke.

PROF. J. W. RICHARDS: Mr. Hirsch's paper on cerium is, to me, an extremely interesting one, because recently I have had occasion to look up this matter of the sparking of cerium alloys. The question has come up as to what causes the sparking of these alloys, and I have put a great deal of attention on it to find out the reason why. The very pure cerium is quite soft, like lead, and when you cut it with a knife, you get a shaving. The cutting off of the shaving does not bring it up to the ignition point. After examining a number of alloys and comparing them with the pure metal, I came to the conclusion that the spark-giving property (which is utilized so largely now in automatic sparking devices) is due to the brittleness of the material, that is, its brittleness in the sense that when it is scraped or scratched by a knife it gives off fine particles. This is not brittleness in the sense that it easily breaks, like chalk, but such that on scratching it with a knife it gives off a spray of fine particles. The energy absorbed in detaching those particles raises them to the ignition point, and then you have the ignition or sparking phenomena. I have experimented with a number of these alloys myself, and I have come to the conclusion that in every case the brittleness as thus defined is the inherent cause of the usefulness of the alloy as a spark-giving metal.

DR. J. W. BROWN: I notice that the alloy of aluminum is stated to be very hard and very brittle, and yet it does not possess this property of giving sparks, according to the theory advanced in your paper. Is this alloy an exception?

MR. A. HIRSCH: In the case of aluminum, I believe that a compound is formed containing about 55 percent of cerium, and this compound does not give the sparks, because in most cases you must have a percentage of about 70 of cerium in order to get this pyrophoric phenomenon. The silicide itself does not give sparks, but if you take that silicide and add to it enough

cerium metal to bring the percentage up to about 70 or 75, you get a very good spark. If you have cerium present to act as a binder, and the percentage of cerium is about 70, you usually get the pyrophoric alloy.

MR. C. A. HANSEN: Can pyrophoric alloys be made by dissolving misch-metal carbide in various metals?

MR. HIRSCH: Cerium carbide is pyrophoric, but it decomposes.

MR. HANSEN: How about iron-cerium carbide?

MR. HIRSCH: In the presence of moisture, this also decomposes.

MR. HANSEN: The alloy, then, would not be permanent?

MR. HIRSCH: It would not.

PROF. RICHARDS: As a matter of scientific accuracy, I would like to call attention to the fact that what we are discussing as a "pyrophoric alloy" is not pyrophoric at all in the true dictionary sense of that term. A pyrophoric material is something which takes fire spontaneously in the air. The use of the term "pyrophoric alloy" originated with Dr. Welsbach when he discovered these sparking alloys, and he used the term "pyrophoric alloys" for the cerium alloys which when scratched gave off sparks. The term "pyrophoric alloy" was thus invented, or used, by Dr. Welsbach in describing these strongly sparking alloys.

I may say that when you start with pure cerium and *increase* the amount of alloying metal, you eventually get a maximum of brittleness. As you increase the amount of alloying metal, you get a greater and greater pyrophoric quality and effect, until at a certain point it reaches a maximum, after which it decreases. That is clearly described in the fundamental Auer von Welsbach patent for pyrophoric cerium alloys.

DR. E. F. NORTHRUP: Did you make observations to find out whether there is any chemical action whatever of the molten cerium on quartz or silica?

MR. HIRSCH: Cerium melts at a temperature of 635°, and when heated 50° to 100° above its melting point, I believe it acts upon quartz.

DR. NORTHRUP: Have you made wire of it?

MR. HIRSCH: No, I have not made wire. That is, I have cut wire, but I have not drawn it with a die.

A paper presented in abstract by Mr. C. A. Hansen, at the Twentieth General Meeting of the American Electrochemical Society, in Toronto, Canada, September 21, 1911, President W. R. Whitney in the Chair.

TRANSFORMATION OF OTHER FORMS OF CARBON INTO GRAPHITE.

By W. C. Arsem.

Introduction.

The conditions under which "Amorphous Carbon" is transformed into graphite have been the subject of much discussion, and many statements have found their way into the literature which are not supported by experimental evidence. The following theories are commonly held:

1. A high temperature alone will convert "amorphous carbon" into graphite (Moissan).

2. Pure carbon is not converted to graphite by heat alone (Berthelot).

3. Graphite is the result of intermediate formation and decomposition of carbides, due to the presence of mineral matter as an original constituent, or purposely added (Acheson).

I have collected all available references relating to this problem, and have given briefly the evidence in favor of each theory.

Despretz[1] heated several varieties of carbon in an arc. He states that "Any carbon which is submitted for a long time to a high temperature becomes proportionately softer. Finally it is transformed into graphite." No physical or chemical tests for graphite are described.

Berthelot[2] was first to recognize the ambiguity in the use of the term "graphite," and proposed the Brodie test as a criterion.

He was not able to convert amorphous carbon to graphite at a *white heat* in hydrogen. A retort carbon rod which had been ignited, thrust into a stream of oxygen, and quenched in water

[1] Comptes Rendus XXIX, 709-724.
[2] Ann. Chim. Phys. XIX, Series 3, 392.

when fully incandescent, was found to have been graphitized on the tip.

Work of Moissan.[3]

Moissan heated in the arc furnace different varieties of carbon enclosed in carbon crucibles, and, after heating, determined their specific gravity and their behavior toward nitric acid and potassium chlorate. He found that the products differed in specific gravity, but in all cases they yielded yellow graphitic acid with the oxidizing mixture. He concluded that heat alone will convert amorphous carbon into graphite. His results are given in the following table:

TABLE I.

Carbon	Ash after firing	Specific Gravity after firing
Sugar carbon	0.11%	2.19
Carbon from Al_4C_3	...	2.11
Cryst. graphite from cast iron, 1150°	1.30	2.17
Cryst. graphite from cast iron, high temperature	0.17	2.18
Cryst. graphite from cast iron cooled in water	1.29	2.16
Graphite from cast iron by silicon	0.85	2.20
Graphite from platinum	1.10	2.06-2.18

He showed also that graphite separates during the cooling of a saturated solution of carbon in various metals, and that graphitic carbon is a product of the decomposition of many carbides, but gives no specific gravity determinations except for Fe and Al.

Work of Acheson.

Acheson noted the formation of graphite by the decomposition of silicon carbide at high temperatures. He also noted that the furnace core of bituminous coal coke in the carborundum furnace became converted, to a considerable extent, into graphite. The following statements, made by Acheson, are to be found in the literature and in patents:

(a) The amount of graphite produced by highly heating pure carbon is insignificant and impracticable, but if the carbon is mixed with considerable mineral matter, the yield of graphite is enormously increased. (U. S. Patent 568,323, Sept. 29, 1896.)

(b) A mixture of 97 parts coke, or charcoal, and 3 per cent. iron oxide, can be changed, to a greater or less extent, into graphite by varying the time and temperature of heating. As

[3] Le Four Electrique.

the iron is insufficient to convert all the carbon to carbide, it is assumed to have a catalytic effect, first forming a carbide which decomposes, yielding graphite and setting free iron, which again forms carbide, and the cycle is repeated. (U. S. Patent 617,979, Jan. 17, 1899.)

(c) A natural variety of carbon containing mineral matter, such as anthracite coal with 5.783 percent ash, gives practically pure graphite with .033 percent ash. This result is assumed to be due to the intimate admixture of "inherent impurities," it being stated that even with the best artificial mixing and distribution of carbon and mineral matter, the conversion to graphite will be more or less irregular and incomplete. (U. S. Patent 645,285, March 13, 1900.)

(d) Petroleum coke, to which has been added 5 percent iron in the form of oxide, is heated to the vaporizing point of iron in a specially constructed electric furnace. The iron vapor is stated to cause conversion of the petroleum coke to graphite. (U. S. Patent 711,031, October 14, 1902.)

(e) Acheson also makes the following statements in an article presented before the Franklin Institute.[4]

1. Comparatively pure petroleum coke produces practically no graphite.

2. Impure bituminous coal coke produces large quantities.

3. The larger the known percentage of impurities in the bituminous coal coke, the greater the amount produced.

4. Only a part of the core in the carborundum furnace (bituminous coal coke) is converted into graphite, this not being increased even by repeated use of the same grains in successive carborundum furnaces. He concludes: "The amount of graphite produced in the core of the carborundum furnace, and also in graphite articles I have made, is much too great to be accounted for by the theory that it is formed by the dissolution of the fixed carbides formed by the contained impurities, and carbon sufficient to satisfy the chemical formula. The most probable and satisfactory explanation is that a catalytic action occurs—a progressive formation and dissolution of carbides. The temperature being much above the point of volatilization of silica and all other

[4] J. Frank. Inst. **147**, 475, 486.

possible impurities, a rapid dissipation of the active agents takes place, and it is completed in this case before the conversion of all the amorphous carbon can occur."

No experimental evidence is given by Acheson to show that pure carbon is not graphitized by simple heating. Direct proof is also lacking to show that a small amount of mineral matter can cause, or facilitate, the conversion of a large amount of carbon to graphite.

The only experiment bearing on this point that I could find described in the literature was in an article by F. A. J. FitzGerald,[5] from which I quote as follows:

"Two carbon rods, one composed of very pure *lamp black carbon,* and containing less than 0.2 percent ash, the other made of *petroleum coke carbon* which had been intimately mixed with a certain amount of ferric oxide, were heated side by side in an electric furnace. At the end of the experiment, the rod that had contained the iron was found to be graphitic; could be easily cut with a knife, took a beautiful metallic luster on rubbing, and would mark paper like an ordinary pencil.

"The pure carbon rod showed little change, was dull black in color, nearly as hard as before heating, and would not leave a mark on paper. One end of this carbon rod, however, was clearly graphite, from the fact that it had been exposed to the action of carbide-forming elements. These vapors had even penetrated to a certain depth in the carbon rod, and in so far as this had occurred the rod showed a brilliant graphitic appearance, was soft, etc."

This experiment would seem to be inconclusive, because two different varieties of carbon were employed, one being heated with iron oxide and the other without. It should have been determined whether or not pure petroleum coke is graphitized when heated without admixture with iron oxide.

Borchers[6] claims that a small amount of Al or Al_2O_3 can convert a considerable quantity of amorphous carbon to graphite.

He does not state what kind of amorphous carbon is used, or what happens if this carbon be heated without addition of mineral matter, but refers to work by Borchers and Mögenburg

[5] J. Frank. Inst. **154**, 338.
[6] Elektrometallurgie (3d ed.) 564 (1903).

("Graphit aus Amorpher Kohler in Borcher's Institut für Metallhuttenwesen und Elektrometallurgie"), which was not available.

Borchers claims priority for the theory that graphite is produced from amorphous carbon by the formation and decomposition of carbides, on the basis of an article in Zeit. f. Elektrochem, *3*, 394 (Mar. 20, 1897), from which he quotes: "Metals which form carbides, alloys or more or less dissociable compounds can assist the crystallization of carbon." The article mentioned was a discussion of methods for the production of the diamond, no reference being made to graphite in this connection.

Moreover, Moissan[7] had previously prepared graphite by crystallization of carbon from solution in metals and by decomposition of many carbides.

Other patents relating to the manufacture of graphite are:

E. G. Acheson, 542,982, July 23, 1895. Purifies carbon by volatilizing impurities in an electric furnace.

H. Y. Castner, 572,472, December 1, 1896. Heats rods of retort carbon or other carbon by current. Product has *lower* density and greater conductivity.

Herbert H. Wing, 598,549, February 8, 1898. Amorphous carbon converted into graphite by prolonged heating at high temperature. (Ordinary coke mentioned.)

Definition of Graphite.

Much confusion has arisen from the uncertainty as to the exact significance of the term "graphite." Many different varieties of graphite have been described, and widely differing values for the various physical constants have been published.

Berthelot was the first to adopt the Brodie[8] test as a criterion. He defined graphite as any variety of carbon which yields graphitic oxide when oxidized with nitric acid and potassium chlorate. Moissan seems to have felt the need of a further criterion, as he defined graphite as a variety of carbon, usually crystalline, having a specific gravity about 2.2, which yields graphitic acid when oxidized.

Some of the uncertainty has been removed by the recent work of Le Chatelier and Wologdine.[9] They showed that a number

[7] Ann. Phys. Chem. (7), 8, 466 (1896).
[8] B. C. Brodie, Liebig's Annalen, **114**, 7, (1860).
[9] Comptes Rendus, **146**, 49 (1908).

of natural graphites, when freed from mineral matter and air, had practically the same specific gravity, 2.255, this value being also obtained for a sample of Acheson graphite.

Charpy[10] considers the density a much better criterion than the Brodie test.

My own work indicates that any variety of carbon, after heating to 3,000°, will give a green or yellow oxidation product by the Brodie test, although many samples have a relatively low specific gravity and lack the physical characteristics of graphite. There is no proof that the yellow oxidation product has the same composition in every case. It may be that a variety of structurally related oxidation-products may exist.

It was found, however, on examination of a large number of carbon samples, that the graphitic properties become more pronounced as the specific gravity approaches 2.26. We have then either a series of carbons of varying molecular complexity, the end member being graphite, or a series of mixtures of graphite with other forms of carbon. In either case it seems to me that there can be little objection to the following definition:

Graphite is that allotropic form of carbon having a specific gravity of 2.25 to 2.26.

Those varieties of carbon which have some of the physical properties of graphite, such as color, softness, and streak, but a lower specific gravity, may perhaps be regarded as impure graphites; that is to say, mixtures of graphite with other forms of carbon.

Purpose of the Investigation.

The points at issue are the following:

1. Can a pure form of carbon be transformed into graphite by simply heating to a high temperature?

2. If this is not the case, is it possible to cause this transformation to occur by heating the carbon, well mixed with a quantity of mineral matter, *insufficient to form carbides* with all the carbon present.

There are two ways of attacking the problem.

(a) To determine the effect of heating various forms of pure carbon alone and with small amounts of added mineral matter.

[10] Comptes Rendus, **148**, 920 (1909).

(b) To remove the mineral matter from certain varieties of impure carbon, which ordinarily change to graphite when heated, and see if the change will still occur.

Both methods were tried.

Experimental Methods.

All samples were ground to fine powder before firing, to facilitate the determination of specific gravity, and all samples were tested, after firing, by treatment with potassium chlorate and nitric acid.

Grinding. To insure freedom from enclosed air which would vitiate the specific gravity results, all samples of carbon were ground in a roller-mill. This is a steel cylinder with a tightly fitting cover on each end, and contains four steel rolls. All the surfaces are case-hardened and polished, to insure a minimum of wear. As to the extent to which iron is introduced, it was found that after 50 hours grinding of a 20 gram sample of petroleum coke, the ash had increased only 0.09 percent. The mill is rotated at 200 r. p. m., and from two to eight hours is sufficient to grind most substances, so that the largest particles are not over .005 mm. in diameter.

Firing. The samples were packed in small Acheson graphite crucibles with tightly fitting covers. These, in turn, were placed in larger tubular graphite crucibles, closed at both ends, the space between the smaller and larger crucibles being packed with Acheson granular graphite, previously fired and containing only mere traces of ash. The samples thus protected were fired at approximately 3,000 to 3,300°C., in an experimental tube furnace for 15 minutes. This furnace consists of a heater tube of Acheson graphite surrounded by an insulated carbon tube with an annular free space between them. The outer carbon tube, is well packed in granular petroleum coke, but no packing material is in contact with the heater. With the exception of the graphite heater tube, which was renewed for each run, the furnace has been used for a great many runs and is practically free from metallic impurities.

When making tests of purified and unpurified samples of the same kind of carbon, the purified samples were always fired in

a tube which had not previously been used for firing impure carbon.

Specific Gravity. Specific gravity determinations were made on samples pulverized as above stated, using a pyknometer of the form shown in Fig. 1, holding about 7 cc. The sample was weighed in the pyknometer, which was then half filled with Kahlbaum's C. P. benzene, and heated to the boiling point of the benzene to remove air and fill the pores of the carbon. Owing to the fineness of the powder, this can be accomplished in a few minutes. The pyknometer was then cooled to 20°C., filled to the mark with benzene, and weighed. Results are referred to water at 4°C. The limit of accuracy is about .005.

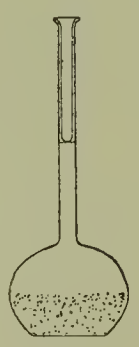

Fig. 1.

Brodie's Test. All samples before and after firing were tested by Brodie's method, which consists in heating the sample with fuming nitric acid and potassium chlorate. "Amorphous Carbon" is converted into brown soluble substances, while graphite is changed to green graphitic oxide, or yellow graphitic acid, according to the concentration of the nitric acid, or the duration of the treatment.

In my experiments I used in some cases nitric acid, distilled from a mixture of concentrated nitric and sulphuric acids. In other cases the nitric acid was made by distilling well dried potassium nitrate with concentrated sulphuric acid, as recommended by Moissan.

Experimental Results.

Petroleum Coke. This was obtained from the Standard Oil Company in lump form, and contained 0.10 percent ash. These lumps, when fired, had all the physical properties of graphite, being soft, and of a silver gray color. They marked paper

easily. A sample of this graphite, pulverized before firing, had, after firing, a specific gravity of 2.26. This value was checked a number of times. A sample of the coke ground with 5 percent ferric oxide had a specific gravity, after firing, of only 2.22.

It is thus seen that quite pure petroleum coke, when fired without additions, yields a very good grade of graphite and that the addition of iron oxide is, if anything, disadvantageous.

Bituminous Coal Coke. This was ordinary foundry coke, containing about 10 percent of red ash, mostly iron and silica.

Lumps of this coke, when fired, became bright gray, and gave the characteristic gray streak on paper. This material seemed harder than petroleum coke graphite. Some of the powdered coke, when fired, had a specific gravity of 2.192.

A sample of unfired coke was treated with fused sodium hydroxide, then with water, and finally with hydrochloric acid. The ash was now reduced to 5.70. After firing, its specific gravity was 2.225.

The results of these experiments are given in Table II.

TABLE II.

	Ash before firing	Ash after firing	Specific Gravity after firing
Crude coke	10.00	2.07	2.192
Treated with fused NaOH, then with HCl	5.70	0.78	2.225

Here we see again the *smaller* the amount of mineral matter present, the *higher* the specific gravity reached on firing.

Anthracite Coal. A sample of Lackawanna coal, containing 6.46 percent ash, when fired in lump form came out bright gray, rather hard, and with a tendency to cleave into plates and angular fragments, like the unfired coal. It marked paper with some difficulty, and its feel and appearance were much less graphitic than in the case of the fired foundry coke and petroleum coke.

A pulverized sample, when fired, had a specific gravity of 2.133.

Some of the pulverized coal, when treated with fused NaOH then washed and treated with HCl, had its ash reduced to 0.20 percent, and this purified coal, when fired had a specific gravity of 2.149, a value slightly higher than that given by the crude coal.

To check this result, some samples of coal obtained from the U. S. Geological Survey were treated in the same way. The results were even more striking, as may be seen from Table III.

TABLE III.

Results of firing anthracite coal above 3,000°.

Coal Samples			Ash before firing	Specific Gravity after firing
Lackawanna		Crude	6.46	2.133
"		Purified	0.20	2.149
U. S. G. S. "Buckwheat No. 1"		Crude	17.68	2.125
"	"	Purified	1.73	2.170
"	" No. 5	Crude	13.30	2.138
"	"	Purified	0.93	2.180

Anthracite coal, therefore, is a form of carbon which graphitizes only imperfectly, in spite of the high percentage of ash which is well distributed throughout the material. We have here, therefore, excellent conditions for catalytic action without a corresponding effect. The results show that the presence of ash *hinders,* rather than *assists* graphitization. Some samples of coal seem to yield purer graphite than others, especially after the ash has been removed.

Lampblack. A commercial variety of lampblack, known as "Patton's Sun-proof," containing about 0.2 percent ash, principally iron oxide, when fired in the tube furnace reached a specific gravity of 2.090. Some of the lampblack, unfired, was ground with 5 percent ferric oxide and fired. This had a specific gravity of 2.094. Another sample of lampblack was impregnated with ferric oxide, so as to obtain as good a distribution as possible in the following way:

The lampblack was stirred into a solution of ferric chloride and ammonia was added to precipitate ferric hydrate, when the lampblack precipitated with the ferric hydrate, forming an apparent homogeneous mixture. This mixture was well dried and divided into two portions; the first was heated one hour in vacuo at 1,600°, and the other one hour at 2,000°, to reduce the iron oxide to metal, and then both were fired in the graphite tube furnace at about 3,300°. After firing, the specific gravity of the

first portion was 2.122, and of the second portion 2.109. All these fired samples were indistinguishable from each other by physical or chemical tests. We have here a pure form of carbon, which reaches a limiting density of about 2.10, and this limit is not appreciably raised by the presence of even 5 per cent ferric oxide, although the distribution of the latter was about as perfect as possible and the conditions especially favorable for a catalytic transformation into a more graphitic variety of carbon if this were possible. They are all grayish-black non-crystalline powders. They all yield by Brodie's test, a yellow graphitic acid, but have none of the characteristic properties of graphite.

Another sample of lampblack, Reichard's No. 7, was also fired, and reached a density of 2.074.

Patton's Sun-proof lampblack was also heated with small percentages of different substances, but no catalytic effect was noticeable.

The results are summarized in the table.

	Specific Gravity After Firing
Patton's lampblack 0.2% ash	2.090
" " + 5% Fe_2O_3 ground together	2.094
" " + 5% Fe_2O_3 by pp n. (a)	2.122
" " + 5% " " " " (b)	2.109
" " + 5% Al_2O_3	2.080
" " + 1% Si	2.076
" " + 5% MnO_2	2.091
" " + 5% NiO	2.099
Reichard's lampblack	2.074

Retort Carbon. The carbon obtained from the inside walls of gas retorts occurs in three varieties, black, gray and white. These differ in ash content and in behavior on heating, and show in a striking way that the ability to graphitize is independent of the amount of ash present. Both the white and the gray varieties yield excellent graphite, although the former is very low in ash and the latter high.

The black variety which is also low in ash does not graphitize but reaches a density of 2.11. Four different substances tried as catalyzers had no effect, the density after firing being practically the same as that of the same carbon fired without mineral additions.

Table IV.

Effect of heating Retort Carbon above 3,000°.

	Ash before firing	Ash after firing	Sp. Grav. after firing
White retort carbon	0.34	0.10	2.265
Gray " "	2.66	0.16	2.263
Black " "	0.23	0.10	2.110
" " " + 1% CaO	..	0.31	2.123
" " " + 1% SiO_2	..	0.12	2.117
" " " + 1% Li_2CO_3	..	0.05	2.120
" " " + 2% Fe_2O_3	..	0.09	2.178

Diamond. Some white diamonds when fired became converted into a coke-like carbon, having a specific gravity of 1.915, which does not mark paper.

Parsons & Swinton[11] heated a diamond in the focus of a cathode ray tube at 1890°, and state that it was converted into coke. The specific gravity was not stated.

These results are especially interesting since it has been commonly supposed that the diamond is converted into graphite by heat, and statements to that effect appear in most text-books.

Conclusions.

All the pure forms of carbon which have been tested, when fired above 3000°, reach a limiting density which is not appreciably raised by the addition of small amounts of mineral matter. The end product is graphite in some cases and not in others. Pure petroleum coke, heated without addition of mineral matter, is converted into graphite of excellent quality, while lampblack, although it increases in density, does not reach the value corresponding to graphite, nor acquire any of its other physical properties, even when heated with various oxides.

The impure carbons show a similar behavior. The properties of these carbons after firing are characteristic for each variety of carbon and independent of the amount of ash present.

Anthracite coal is only imperfectly graphitized by heating. The specific gravity of the fired material was approximately the same for three samples having a range of ash content from 6.46 percent to 17.68 percent. Moreover, coal from which most of the ash has been previously removed graphitizes better than the crude material.

[11] Proc. Roy. Soc. **80**, 184, (1908).

Bituminous coal coke, which graphitizes fairly well, yields an even better product if a part of its ash has been removed before firing.

It must therefore be concluded that a small amount of mineral matter exercises no beneficial effect in the manufacture of graphite by the heating of carbon, and that the quality of the product cannot be improved in this way. As to the effect of mineral matter on the rate of conversion, no data are yet available, although some experimental work along this line has been started. At 3000° the maximum density is reached in less than 15 minutes for any variety of carbon.

We need some theory of the nature of graphite and of amorphous carbon which will permit a rational explanation of the changes which occur on heating. The following discussion is a tentative step in this direction.

The Nature of Graphite.

Graphite, in the most restricted sense of the term, is an allotropic form of carbon having a definite and not very complex molecular configuration. The molecule might, for example, be regarded as two benzene rings side by side and joined at all six angles, the extra bonds being satisfied as in the usual centric formula. Such a formula might be used to explain the formation of Mellitic acid and graphitic acid by oxidation of graphite.

Amorphous Carbon. When an organic compound is decomposed, there results a mixture of substances constantly increasing in complexity until finally carbon is obtained. This carbon need not be regarded as a simple substance, but may be considered to be a mixture of many varieties of carbon, each with a different number and arrangement of atoms in the molecule. According to this view, it seems better not to regard "Amorphous Carbon" as the name of a distinct allotropic form, but rather as a general term covering all varieties of carbon except graphite and diamond. In speaking of amorphous carbon, therefore, the source of each sample should be stated.

The Mode of Transformation. In a given sample of amorphous carbon, some of the molecules will be capable of easily undergoing rearrangement under the influence of heat to form

graphite molecules, while others will not, and the proportion of molecules capable of such change will determine the character of the final product.

Petroleum coke would therefore consist almost wholly of graphitizable molecules, while anthracite coal contains a smaller proportion.

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DISCUSSION.

PRESIDENT WHITNEY: Perhaps no man in this country has worked so much on carbon and graphite as Mr. Arsem. He has been working at it for a matter of six or eight years to my certain knowledge, and we ought to read with great care and attention what he says about it. Graphite is difficult to define, and yet we are always trying to define it. Nature does not permit us to define very accurately and maintain our definitions over long periods of time. As, probably, you all know, and as Mr. Arsem points out, it has been customary to recognize the Brodie test as the criterion of graphite.

That form of carbon which has an "unctious feel," marks paper with a "graphitic" streak, and when heated in a mixture of anhydrous nitric acid and potassium chlorate gives an insoluble yellow or green graphitic oxide, is called graphite.

Mr. W. C. Arsem has defined graphite as "that allotropic form of carbon having a specific gravity of 2.25 to 2.26." Although we are not obliged to accept that as our definition, yet it does bring our experimental data up to date, so to speak.

MR. C. A. HANSEN: Some years ago, at the suggestion of Dr. Weintraub, I prepared some very peculiar graphite by reversing the current in the usual mercury arc. In this arc, mercury is normally the cathode, and Acheson graphite the anode, and no disintegration of the graphite takes place. With the graphite as cathode, the cathode spot meanders up and down and

around the graphite, leaving a worm trail; and an extremely fine powder is thrown off, so fine, in fact, that it will remain suspended for hours in ether.

When carefully freed from mercury by nitric acid, and thoroughly washed, this powder responds to the Brodie test in a single treatment.

An attempt was made to press this powder into a rod by submitting it to a pressure in a plunger and die arrangement of over 50 tons per square inch. Here it became evident that the powder had none of the lubricating and cohesive properties of ordinary graphite, for the plunger shrieked while being forced into the die, and after the pressure was removed the rod again became a loose powder.

While pressing fine Acheson graphite powder in the same die, the plunger enters smoothly and easily, and the resulting rod, when broken up, resolves itself into more or less flat sheets somewhat resembling natural graphite.

Scarcely enough of the peculiar graphite or carbon powder was available for a specific gravity test.

A paper read by Dr. W. D. Bancroft at the Twentieth General Meeting of the American Electrochemical Society, in Toronto, Canada, September 21, 1911, President W. R. Whitney in the Chair.

THE EFFECT OF ORGANIC AND INORGANIC "ADDITION-AGENTS" UPON THE ELECTRO-DEPOSITION OF COPPER FROM ELECTROLYTES CONTAINING ARSENIC.*

By Ching Yu Wen and Edward F. Kern.

Introduction.

In the production of good and pure copper by electrolysis, the composition of the electrolyte is one of the important factors. The causes of poor copper deposits are chiefly the impurities which accumulate in the electrolyte, and which, under usual conditions, are precipitated along with the copper. Of those impurities, the most harmful and troublesome ones are arsenic and antimony, the presence of which in the deposited copper makes it brittle and nodular.

It has been a known fact that during electrolysis part of the arsenic and antimony contained in the anode dissolves and remains in solution. These two elements, especially the arsenic, are allowed to accumulate in the electrolyte till a critical point is reached, which has not yet been definitely determined. When this point is passed they begin to be deposited with the copper on the cathode and render the deposit impure and brittle. To prevent this, it is therefore of utmost importance to maintain the electrolyte within a certain degree of purity, in other words, to keep the amount of arsenic and antimony in the electrolyte below the danger line. This is usually accomplished in practice by withdrawing a certain portion of the electrolyte and replacing it with an equal quantity of fresh solution, and the copper in the impure electrolyte is recovered either by crystallization or by electrolysis with insoluble lead anodes. This not only complicates the process of electrolytic refining of copper, but also entails an extra item of expenditure in the production of electrolytically refined copper.

* This article is based upon investigations conducted in the Metallurgical Laboratory of the School of Mines, Columbia University, by Dr. C. Y. Wen.

Another thing that is observed during the electrolysis is that "sprouts" or dendritic "trees" often form, especially along the edges of the cathode. The formation of such "trees" interferes with the work, renders it more difficult, and prevents the electrodes from being placed close together, as there is danger that the electric current will be short-circuited. In conducting the electrolysis in a commercial way, the removal of these "trees" becomes absolutely necessary, and is usually done by the tank inspectors, thus increasing the cost of refining.

The object of the investigation was, therefore, two-fold: First, to find conditions which would prevent the deposition of arsenic and antimony on the cathode, and, second, to prevent the formation of dendritic "trees." This problem was worked out, having in mind the production of commercially pure copper as solid and smooth deposits, from copper electrolytes containing high percent of arsenic, by the use of organic and inorganic "addition-agents."

ABSTRACTS OF LITERATURE.

In reviewing the literature regarding both organic and inorganic addition-agents, little was found.

Kiliani[1] was, perhaps, the first who conducted systematic experiments to investigate the behavior of impurities present in the copper anode and to study the effect of inorganic salts on the character of the copper deposit. For the latter case he used an electrolyte containing 15 grams of copper sulphate and 5 grams of sulphuric acid in 100 c.c. solution, with a current density of 20 amperes per square meter. He observed the fact that with a small amount of tin salt in the electrolyte, good, smooth, malleable copper was produced, while in the case where the electrolyte contained no tin salt the deposit was extremely bad and brittle. He noted also the fact that the presence of a small amount of tin in the anode caused the potential difference between electrodes to be greatly reduced.

W. Borchers[2] performed experiments with the object of preventing the crystalline growth of copper on the deposit by adding to the electrolyte a sufficient amount of sodium chloride, or magnesium chloride. He found, however, that by the addition of these reagents only a diminution of the evil could be effected.

[1] Berg und Hüttenmannisches Zeitung, 1885, p. 249.

[2] W. Borchers, "Electrolytic Smelting and Refining," p. 206 (translated by McMillan).

H. O. Hoffman[3] has pointed out that hydrochloric acid is used in practice to precipitate the antimony in the electrolyte. This is accomplished by the addition of a sufficient quantity of crude hydrochloric acid to the head tank to maintain 0.04 gram of chlorine per liter in solution. The hydrochloric acid reacts with the antimony and precipitates it as oxychloride. When there is a deficiency of hydrochloric acid the cathode becomes streaked, tarnished, black and brittle.

It is said that ammonium sulphate[4] has been used in the electrolyte to hinder the precipitation of arsenic on the cathode, and the amount usually added was from 0.5 to 20 percent. The addition of this salt decreases the conductivity of the electrolyte.

L. W. Wickes[5] investigated the percent of arsenic which the deposited copper would contain for a given potential between electrodes, and the relation between variations in the potential and the amount of arsenic in the copper deposited. For his experiments he used anodes containing 1 percent, 2 percent and 4 percent arsenic, and an electrolyte containing 75 parts of water, 19 parts of copper sulphate and 6 parts sulphuric acid, by weight, and also an electrolyte of the same composition, but containing 0.101 percent arsenic in the form of arsenic acid. The experiments were conducted with 0.4 volt, 0.6 volt and 0.8 volt. He found that, with different voltages and the same percent of arsenic in the anode, the percent of arsenic in the cathode copper was practically the same in all cases, and that the greater the percent of arsenic in the anode the more erratic were the results. The conclusion which he drew from his experimental data was that the percent of arsenic in the deposited copper is not a function of the potential between electrodes, but of the degree of hydrolyzation of the sulphate of arsenic in the electrolyte.

Of the work on organic addition-agents, that of Edward F. Kern and Royal P. Jarves[6] should be mentioned. They conducted experiments on the effect of the presence of tannin, pyrogallol, gelatine and resorcinol upon the density and coherence of electrolytically deposited copper, lead and silver. For

[3] T. A. I. M. E., 34, 312 (1904).
[4] T. Ulke, "Modern Electrolytic Copper Refining." 1st ed., p. 18.
[5] E.M. thesis, Metallurgical Library, School of Mines, Columbia University.
[6] School of Mines Quarterly, 30, 119 (1909).

their experiments on copper they used two kinds of electrolyte, the cupric sulphate and the cupric fluo-silicate. With the former electrolyte, which contained 16 grams of cupric sulphate ($CuSO_4.5H_2O$) and 4 grams of sulphuric acid (H_2SO_4) per 100 c.c., they found that the presence of tannin, gelatine or resorcinol equally caused the copper to deposit more smoothly. The deposited copper formed at 30° C. was better than at 20° C. With the fluo-silicate electrolyte, which contained 6.34 grams of copper and 3.60 grams of free hydro-fluo-silicic acid (H_2SiF_6) per 100 c.c., they observed that the deposits were rendered brighter and more smooth by the presence of tannin, gelatine or pyrogallol. The first of these addition-agents was the most effective, while the last two were somewhat less. Better results were also obtained at 30° C. than at 20° C.

In regard to the function of organic addition-agents, Edward F. Kern[7] has also performed experiments, for which the following electrolytes were used:

1. Copper electrolytes, which consisted of cupric sulphate, cuprous chloride and cupric fluo-silicate.
2. Lead electrolytes, which consisted of lead nitrate and lead fluo-silicate.
3. Silver electrolytes, which consisted of silver nitrate and silver fluo-silicate.

The organic addition-agents employed were gelatine, resorcinol, pyrogallol and tannin. From the results of his experiments he concluded as follows: "That the most suitable organic addition-agents for copper, lead and silver electrolytes are compounds of the benzene ring series, which have a large number of adjoining hydroxyl radicals; and, also, the greater the molecular weight of the addition-agent, in other words, the larger the numbers of hydroxyls, the more effective it is in producing more satisfactory results."

"If it is the hydroxyl (and, as well, the amine) radicals of organic addition-agents which cause deposits to form denser, smoother and less crystalline, then no doubt this effect may be attributed to the reducing property of the addition-agents. This statement was suggested by recalling a general rule of organic

[7] Tran. Amer. Electrochemical Society, **15**, 441-476 (1909).

chemistry, which is: 'The most easily oxidizable organic compounds of the benzene ring series, and those which more readily precipitate metals from their solutions, are compounds which contain the largest number of hydroxyl or amine radicals; and the compounds which are most easily oxidized are those in which the hydroxyl and the amine radicals are the more closely grouped.'"

From his experimental data and the above generalizations he has deduced a theory regarding the function of an addition-agent, which reads: "The function of an addition-agent in an electrolyte is to maintain a reducing menstruum around the cathode, which, in turn, causes the electro-deposit to form denser and smoother."

"The fact that the consumption of organic addition-agents is in proportion to the amount of metal deposited is an evidence of their reducing action. And, for this reason, in order to maintain the deposition of smooth, dense, coherent deposits, the organic addition-agent must be added in definite amounts to the electrolytes from time to time."

From the results of his investigations on inorganic addition-agents,[8] he writes as follows: "The deposits formed in electrolytes which contain alkali or alkaline earth salts are generally denser, smoother and less crystalline than those which are formed in electrolytes which do not contain these salts. This is the case with nickel sulphate and nickel chloride electrolytes, which contain salts of sodium, potassium or magnesium. The formation of smoother and less crystalline deposits from these electrolytes may be attributed to the reducing action of the sodium, potassium or magnesium ions in the layer of electrolyte which surrounds the cathode. Ammonium salts act similarly to the alkali salts, but to a less marked degree. These facts also seem to conform to the above advanced theory."

Not only have the organic and inorganic addition-agents been found to improve copper deposits, but the temperature of the electrolyte has also been found to exert a marked beneficial influence. The investigations of Förster and Seidel[9] have shown that the deposits produced at 40° C. were uniformly crystalline and possessed great ductility, and those formed at 60° C. were less ductile and of coarser crystals. They have also shown that the

[8] Trans. Amer. Electrochemical Society, **15**, 472 (1909).
[9] Zeitschrift für Electrochemie, **5**, 508 (1899).

deposits formed at higher temperatures were of greater tensile strength than those formed at lower temperatures.

There was found other reference to addition-agents in electrolytes of lead, silver and nickel, but as these have nothing to do with the present investigation, they will not be here discussed.

EXPERIMENTAL PART.

Preparation of Copper-Arsenic and Copper-Antimony Alloys.

For making the anodes, alloys of copper-arsenic and of copper-antimony were first prepared. Ten pounds (4.5 kg.) of granulated copper, covered with a layer of charcoal, were first melted in a Dixon graphite crucible (No. 20) in a gas-fired furnace. When the copper was completely melted, it was thoroughly "poled" with sticks of wood. The crucible was then taken out of the furnace, and to the molten copper 1.5 pounds (0.68 kg.) of metallic arsenic, wrapped with copper foil, was added. The crucible was again heated. The molten alloy was stirred with a graphite rod so as to secure uniform composition. It was granulated by pouring slowly at a vertical distance of six or seven feet (1.8 to 2.1 m.) into a large basin full of cold water 2 feet (0.6 m.) deep and 2½ feet (0.75 m.) in diameter. The copper-antimony alloy was made in the same way as above, except that 0.5 pound (0.23 kg.) of antimony was added in small chunks to 10 pounds (4.5 kg.) of molten copper.

Making of Anodes.

Twelve pounds of granulated refined copper, covered with a layer of charcoal, were first melted in a Dixon graphite crucible in a gas-fired furnace and "poled" to tough-pitch, as described in the case of making copper-arsenic alloy. Then 1.5 pounds (0.68 kg.) of the copper-arsenic alloy and 2 pounds (0.91 kg.) of copper-antimony alloy were mixed and added to the molten copper. Having been thoroughly stirred with a graphite rod, it was cast into anodes in an iron mould, which had previously been warmed and painted with an emulsion of water and bone ash. The size of the anodes was 4¼ inches (11 cm.) high, 2¾ inches (7 cm.) wide and ⅜ inch (9.5 mm.) thick.

A small portion of the anode copper was granulated as before, and taken for analysis for arsenic and antimony.[10]

Preparation of Electrolytes.

Two standard electrolytes were prepared for the electrolyses:

1. Electrolyte A, which contained 15 percent $CuSO_4.5H_2O$ and 10 percent H_2SO_4, by weight, per 100 c.c. solution.

2. Electrolyte B, which contained 15 percent $CuSO_4.5H_2O$, 10 percent H_2SO_4 and 10 percent As in the form of H_3AsO_4, by weight, per 100 c.c. solution.

For making electrolyte A, 160 grams of technical bluestone crystals from Eimer and Amend, New York, were weighed out and dissolved in about 700 c.c. of water. When solution was complete, 63 c.c. of concentrated sulphuric acid (100 grams H_2SO_4) were added to it, and the solution, when cooled, was diluted to exactly 1,000 c.c. The electrolyte was analyzed for copper and free sulphuric acid, and found to contain 15.01 grams of $CuSO_4.5H_2O$ and 9.92 grams of free sulphuric acid per 100 c.c. solution.

The electrolyte was found, by this method, to contain 9.92 grams of free sulphuric acid per 100 c.c. solution.

The preparation of electrolyte B consisted in dissolving 100 grams of metallic arsenic in a sufficient quantity of concentrated nitric acid in a No. 5 beaker, much excess being avoided. After complete dissolution of arsenic, 63 c.c. of sulphuric acid was added, and the solution was heated on a hot plate until the arsenic acid separated out and became a white, pasty mass, which indicated complete expulsion of nitric acid. The arsenic acid was taken up with 200 to 300 c.c. of water; at the same time 160 grams of technical bluestone crystals were dissolved in about 300 c.c. of water in another beaker. The two solutions were mixed and diluted to exactly 1,000 c.c. The arsenic in this electrolyte was determined as follows: 10 c.c. of the electrolyte was measured out and diluted to exactly 500 c.c. in a calibrated flask, from which 50 c.c. was drawn for analysis. It was evaporated to sulphuric fume with an addition of 8 c.c. of concentrated sulphuric acid. The reduction, distillation and titration were conducted in the same way as in the case of determining arsenic

in cathode copper.[10] The analyses showed that the electrolyte contained 10.01 and 9.91 grams arsenic in 100 c.c. of solution.

The electrolytes used for electrolysis were made to contain 1.5 percent, 2 percent, 3 percent, 4 percent, 6 percent and 8 percent of arsenic, prepared from the above two standard electrolytes, by mixing as follows:

(*a*) For electrolyte containing 1.5 percent arsenic, 10 percent free H_2SO_4 and 15 percent $CuSO_4.5H_2O$, 850 c.c. of electrolyte A was mixed with 150 c.c. of electrolyte B.

(*b*) For electrolyte containing 2 percent arsenic, 10 percent free H_2SO_4 and 15 percent $CuSO_4.5H_2O$, 800 c.c. of electrolyte A was mixed with 200 c.c. of electrolyte B.

(*c*) For electrolyte containing 3 percent arsenic, 10 percent free H_2SO_4 and 15 percent $CuSO_4.5H_2O$, 700 c.c. of electrolyte A was mixed with 300 c.c. of electrolyte B.

(*d*) For electrolyte containing 4 percent arsenic, 10 percent free H_2SO_4 and 15 percent $CuSO_4.5H_2O$, 600 c.c. of electrolyte A was mixed with 400 c.c. of electrolyte B.

(*e*) For electrolyte containing 6 percent arsenic, 10 percent free H_2SO_4 and 15 percent $CuSO_4.5H_2O$, 400 c.c. of electrolyte A was mixed with 600 c.c. of electrolyte B.

(*f*) For electrolyte containing 8 percent arsenic, 10 percent free H_2SO_4 and 15 percent $CuSO_4.5H_2O$, 200 c.c. of electrolyte A was mixed with 800 c.c. of electrolyte B.

The Electrolysis.

The electrolyses were conducted in No. 6 beakers (size 4½ inches = 11.5 cm. in diam. and 5¾ inches = 15 cm. high) in each of which was immersed an anode and a cathode (size 4⅜ inches = 11 cm. long and 2¾ inches = 7 cm. wide), the latter being plates cut from sheet copper 1/64 inch thick. The anodes were cast plates of copper ⅜ inch (9.5 mm.) thick. Before the cathodes were used they were straightened and cleaned by washing first with a little dilute nitric acid and then with water, which gave a clean, bright surface. A horizontal mark was made on

[10] The methods which were used for the determination of arsenic and antimony in the anode and cathode copper and in the electrolytes were published in "The Metallurgical and Chemical Engineering" IX, July 1911, pp. 365-367.

each, 4 inches (11 cm.) from the bottom, so as to obtain the desired current density, and the surfaces were lightly greased with vaseline. The anode and the cathode in each cell were suspended from glass rods (*e*, Fig. 1) at a distance of 1¾ inches (4.9 cm.) and parallel to each other. The current density used in all experiments was 40 amperes per square foot (4.4 amperes per sq. dm.).

The Apparatus.

The apparatus and its arrangement are shown in Fig. 1. It consisted of two water-baths (*W*), used to keep the electrolyte at constant temperatures, in each of which were placed four cells (*C*) (No. 6 beakers). Each cell rested on two strips of wood, *s*, and the water-baths were supported at each end by an iron tripod (*I*). Under, and in the middle of each water-bath, was placed a Bunsen burner (*B*), by which the desired temperatures of the two baths could be secured and adjusted. All the cells were connected in series. The electric current used for electrolysis was furnished by a storage battery of six cells, connected in series, measured by the ammeter (*A*), which permitted reading to 0.05 ampere, and regulated by the rheostat (*R*).

Circulation of Electrolyte.

The electrolyte in each cell was agitated during electrolysis by stirrer (*g*). Fig. 1 shows its arrangement and details. It consists of the framework of which (*F*) are the two horizontal steel bars, and the ends of which are clamped to and supported by a vertical iron rod (*L*). To each of the steel bars (*F*) are clamped four cylindrical collars (*h*), at such distance as to conveniently permit the stirrers to operate in the cells. The pulleys (*p*) (⅞ inch = 2.2 cm. diam.), fastened to the stirring rods (*f*), rest on the collars (*h*). The upper part of the stirring rod (*f*) is of steel and the lower part (*g*) of glass rod, and these two parts are connected by means of a short piece of rubber tubing (*r*). Two rubber "policemen" (*b*) attached to the glass rod, as are shown in Fig. 1, serve to give an effective circulation of the electrolyte in each cell. The stirrers are run by a half horse-power motor (*M*). The speed of the stirrers was about 120 revolutions per minute.

Mode of Operation.

In these experiments the mode of operation was simple and may be stated as follows: After all the connections of the circuit had been made, and the electrodes had been properly placed and adjusted, the cells were filled with electrolyte until its surface reached the horizontal mark of the cathodes, which, as has already been said, were scratched for the purpose of obtaining the desired

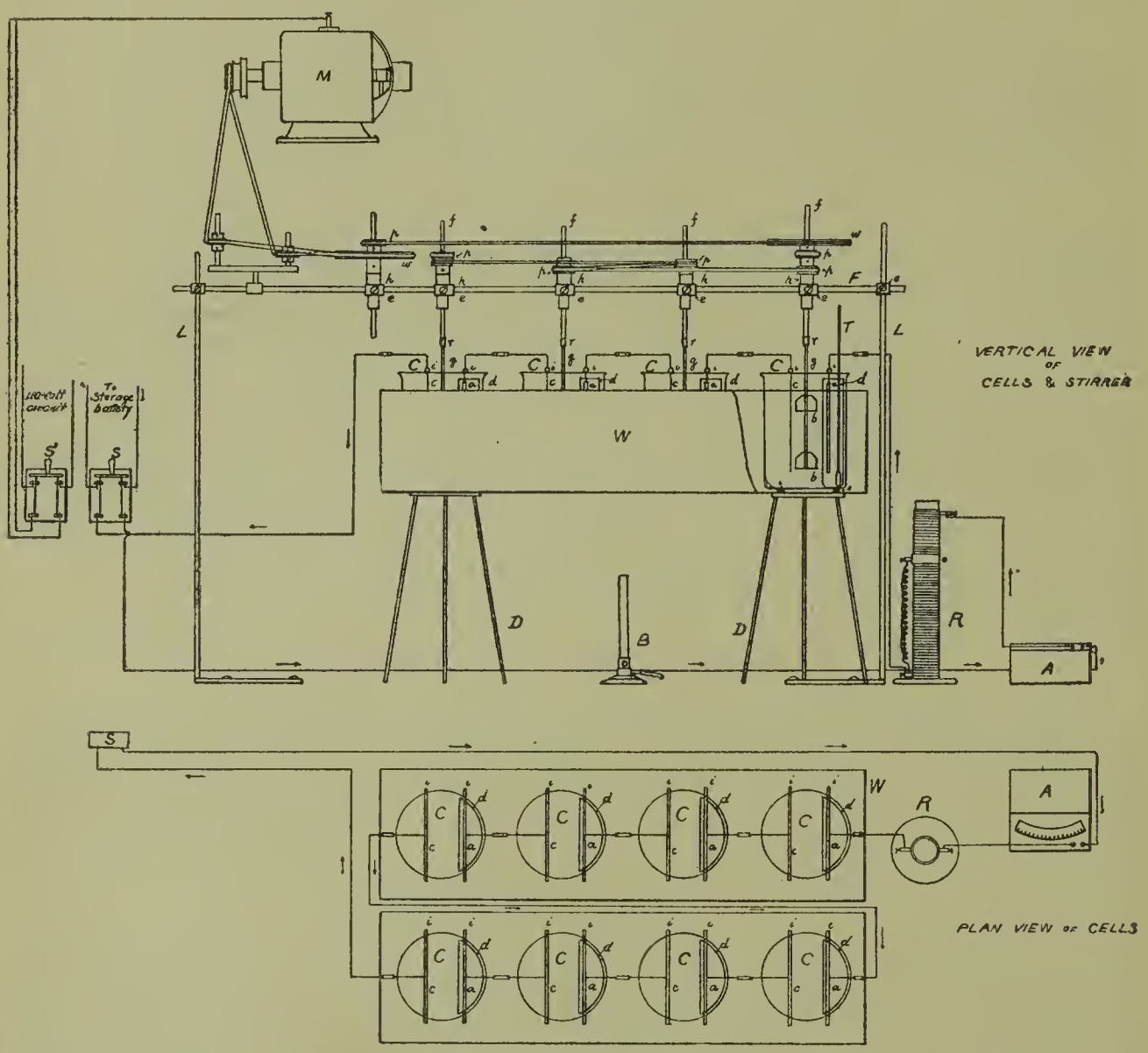


FIG. 1.

current density and which also served to indicate a constant volume of the electrolyte. Water was then run into the two baths (*W*) until they became full. The stirrers were set in motion, and the baths (*W*) heated. When the desired temperatures of the electrolytes were attained, the electric current for electrolysis was turned on and kept constant by regulating the rheostat (*R*). The difference of potential between the anode and

the cathode was read every two hours and sometimes every three hours, with a voltmeter which permits reading to 0.01 volt. The temperatures of the electrolyte were measured with thermometers (*T*) immersed in the cells. At the end of the experiment the glass part of the stirrers was disconnected. The cathode copper was removed from the cells, washed and dried.

During electrolysis it was observed that much water of the electrolyte was lost by evaporation. To make up this loss, water was added to the cells from time to time.

Sample of Cathode Copper for Analysis.

In order to easily peel off the starting sheet, the edges of the cathode were first sawed off, and the deposited copper was sawed into strips from ½ to ¾ inch (1.3 to 1.9 cm.) wide and from 3 to 3½ inches (7.5 to 9 cm.) long. These strips were used as samples for analysis, usually about 80 grams, unless the copper deposit showed dark surface and was brittle, indicating much impurities; in the latter case a sample of 40 grams was taken.

Electrolytes, which Contained no Addition-Agent.

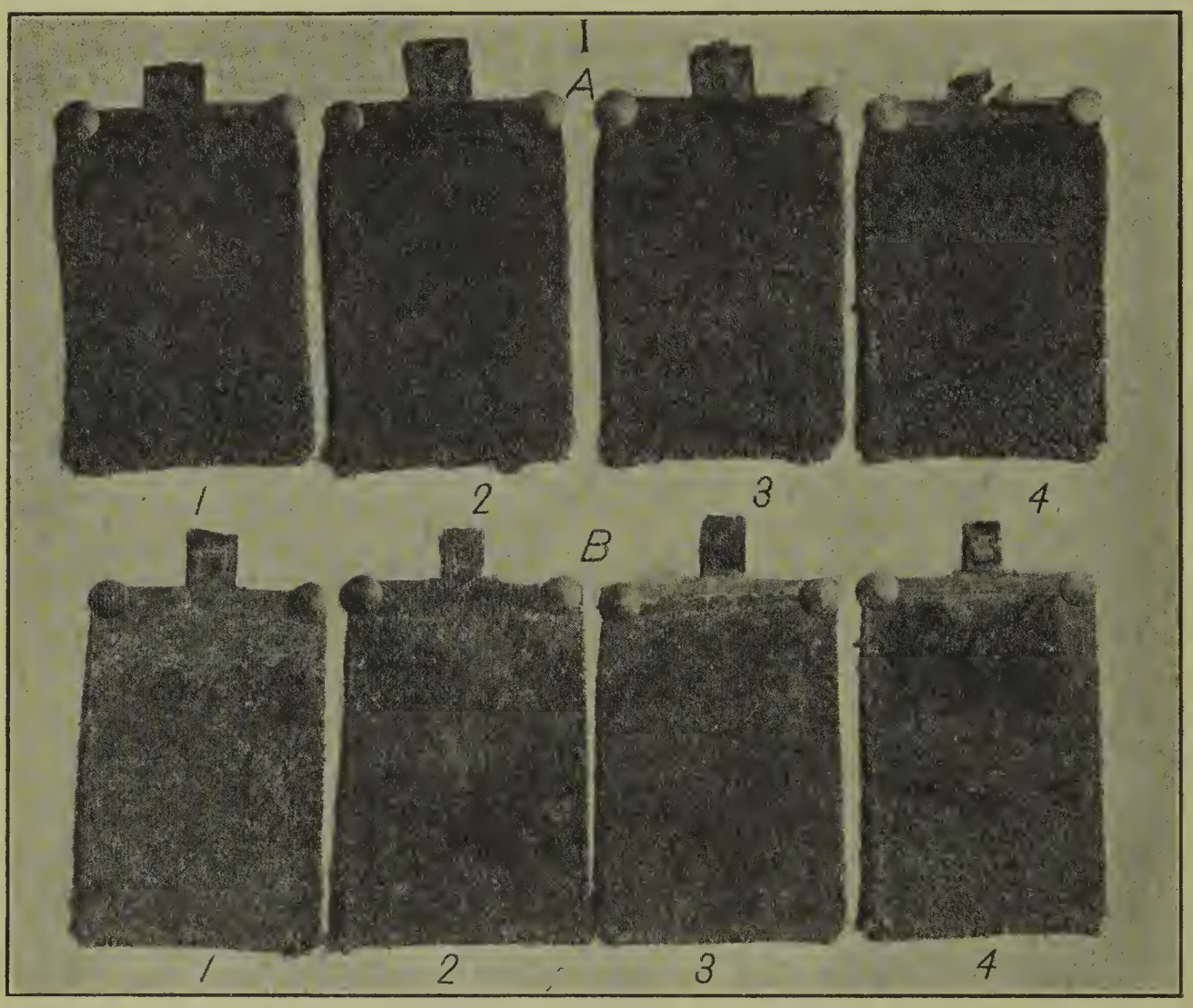
Experiment I.

Four runs were made on electrolytes which contained no "addition-agent." Electrolytes used for Experiment I contained 15 percent cupric sulphate ($CuSO_4.5H_2O$), 10 percent sulphuric acid; and cell 1, no arsenic; cell 2, 1 percent arsenic; cell 3, 2 percent arsenic; and cell 4, 4 percent arsenic. The electrolysis was conducted for fifty-three hours, in two series, each of which consisted of four cells, *C*, as is shown in Fig. 2. The composition of the corresponding electrolytes in the two series was the same, but the temperature of one series was 20° C. and of the other 50° C.

It was observed during electrolysis that the potential difference between the electrodes in the lower-temperature series gradually increased; and that in the higher-temperature one only to a small extent, and the others remained practically constant. The increases of voltage was due to the fact that a sticky coating of oxides of arsenic and antimony gradually formed on the surface

of the anode. But the coating which formed at a higher temperature was porous, offered little or no resistance, and dropped to the bottom of the cell when sufficiently thick. In order to prevent the accumulation of the coating on the anodes in the lower-temperature series, the surfaces were occasionally scraped.

It may be here mentioned that an attempt was made to run this experiment continuously, in other words, day and night; but it was soon realized that this could not be done because two



of the conditions could not be properly maintained during the night. In the first place, the coating of arsenic and antimony oxides which formed on the surface of the anodes in the lower-temperature series was so thick that it greatly increased the resistance, thus reducing the current density; and, in the second place, the addition of water to the electrolytes to make up the loss by evaporation could not be readily accomplished. The result was that the hotter electrolytes became more concentrated. As the deviations of these conditions might have exerted

a strong influence upon the physical and chemical properties of the deposit, analyses of the cathode copper for arsenic and antimony were not made, nor were these two elements determined in the electrolyte after this run.

In regard to the character of the deposits, a few words, however, may be said. It was observed that the deposits formed at 50° C. were, by far, better than those formed at 20° C. They were solid, coherent, and of bright color, though nodular crystals formed at the lower edge, especially in electrolytes containing higher percentage of arsenic. The deposits which formed at 20° C. in electrolytes containing 1 percent, 2 percent and 4 percent were dark, brittle and crystalline, and the crystals were easily broken off. The deposit formed at 20° C. in electrolyte which originally contained no arsenic appeared bright. The deposits are shown in Photograph I. The upper row (A) are deposits formed at 20° C., and the lower row (B) are deposits formed at 50° C.

TABLE I.

Time of experiment, 53 hours. Current density, 40 amp. per sq. foot (4.4 amp. per sq. dm.). Distance between electrodes, 1.75 inch (4.5 cm.). Electrolyte contained 15 percent $CuSO_4.5H_2O$ and 10 percent H_2SO_4. Anode contained 1.15 percent As and 1.05 percent Sb.

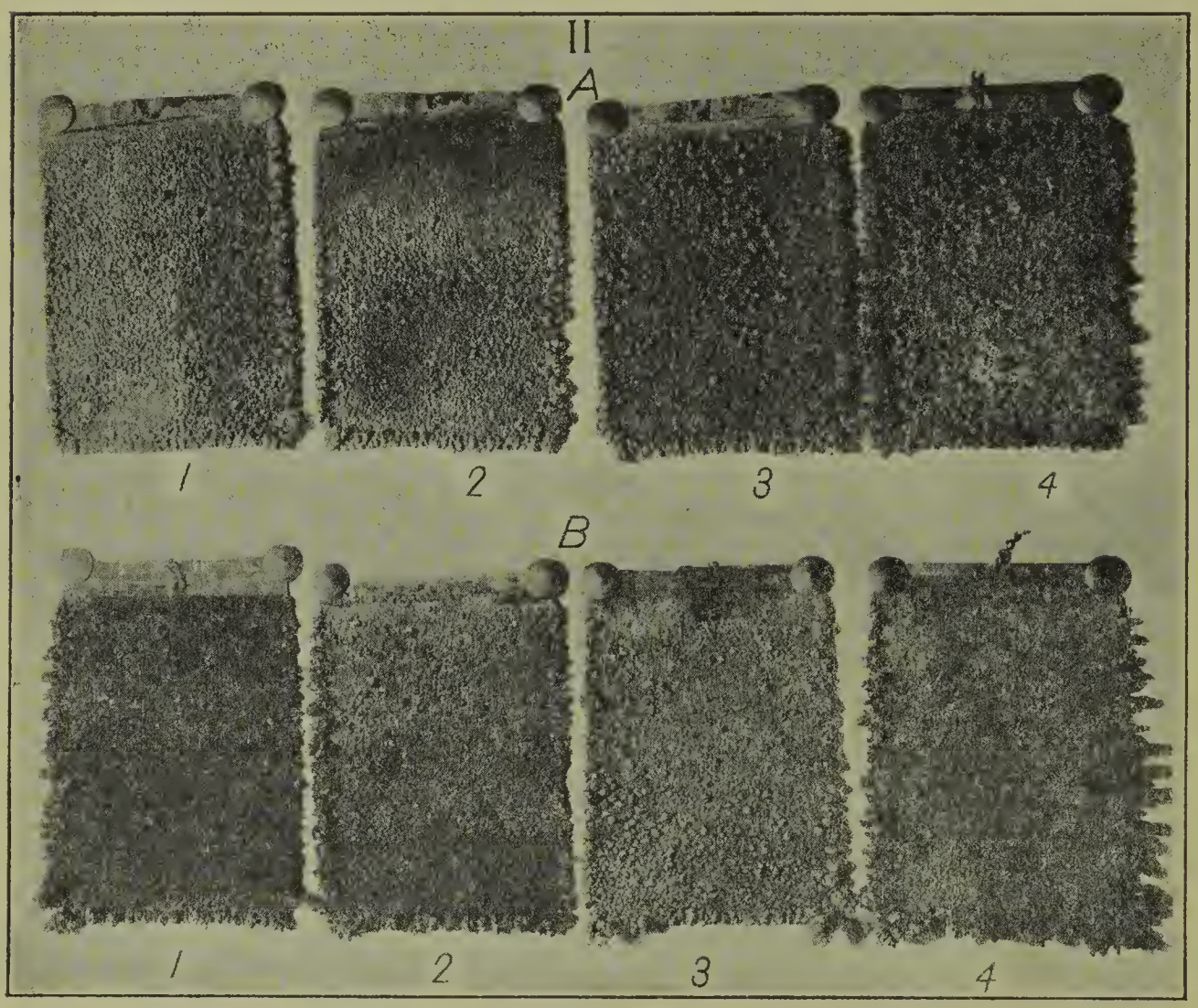
No. of Cells	Percent of As in Electrolyte	Temp. of Electrolyte Degrees Cent.	Photo I
Series A			A
1	0.0	20	1
2	2.0	20	2
3	4.0	20	3
4	4.0	20	4
Series B			B
1	0.0	50	1
2	1.0	50	2
3	2.0	50	3
4	4.0	50	

Experiment II.

This experiment was conducted in the same manner as the previous one except that the electrolysis was carried on only during the daytime. The electrolytes used for this experiment contained 2 percent, 4 percent, 6 percent and 8 percent of arsenic with the usual proportion of cupric sulphate (15 percent $CuSO_4$.

$5H_2O$) and free sulphuric acid (10 percent H_2SO_4). The temperature of the two series were 35° C. and 60° C., and the time of the experiment was forty-two hours.

The deposits former at 35° C. and in electrolytes containing 2 percent, 4 percent and 6 percent arsenic were bad and brittle and composed of coarse grains which were easily detached. Their surfaces became dark as soon as they were taken out of the electrolytes and exposed to the air. The analysis shows that



these deposits were high in arsenic and antimony (see Nos. 1, 2 and 3, Table II). The deposit formed in electrolyte containing 8 percent arsenic and at the same temperature (35° C.) was much better. It was good, bright and coherent, and ran low in arsenic and antimony (see No. 4, series A, Table II). But it was observed that at the edge of this deposit dendritic nodules formed, and smaller nodules were scattered over the surface. This is clearly shown in series A, Photograph II. The deposits formed at 60° C. and in electrolytes which contained 2 percent

and 4 percent arsenic were also bad, brittle, crystalline and incoherent. "Trees" were formed at the edges, as shown in 1 and 2, series B, Photograph II. Their surfaces were dark, and became darker when exposed to air. They ran much higher in arsenic and antimony than those formed at the lower temperature and in similar electrolytes. Those formed at 60° C. but in electrolytes containing 6 percent and 8 percent arsenic were, on the other hand, good, bright, solid and coherent, though larger "trees" were found at the edges, especially of the deposit formed in electrolytes containing 8 percent arsenic; 3 and 4, series B, Photograph II, show the "trees" of these deposits.

The analysis of the electrolytes after the run, as given in Table II, shows that they contained higher percentage of arsenic than before the run. This increase was undoubtedly due to the partial dissolution of the arsenic in the anode. The antimony, both in the cathode copper and in the electrolyte, was also transferred from the anode by the current.

TABLE II.

Time of experiment, 42 hours. Distance between electrodes, 1.75 inch (4.5 cm.). Electrolytes contained 15 percent $CuSO_4.5H_2O$ and 10 percent H_2SO_4. Anode contained 1.15 percent As and 1.05 percent Sb. Current density, 40 amp. per sq. foot (4.4 amp. per sq. dm.).

Number of Cells	Percent of As in Electrolyte	Temp. of Electrolyte in Degrees Cent.	Average E. M. F. in Volt	Impurities in Deposited Copper		Impurities in Elect. after Run		Photo II
				Percent As	Percent Sb.	Percent As	Percent Sb.	
Series A								A
1	2	35	0.58	0.199	0.253	2.22	0.004	1
2	4	35	0.57	0.217	0.391	4.32	0.002	2
3	6	35	0.53	0.314	0.361	6.15	0.003	3
4	8	35	0.59	0.036	0.014	8.15	0.004	4
Series B								B
1	2	60	0.45	0.026	0.395	2.29	0.006	1
2	4	60	0.42	0.388	0.496	n.d.	n. d.	2
3	6	60	0.39	0.018	0.009	6.23	0.002	3
4	8	60	0.38	0.014	0.004	8.10	0.004	4

The potential difference between the electrodes should also be mentioned. It was observed that the potential difference in series A was higher on the first day than on the last day of the run, while that in series B was not so in every case. The potential

difference in cells 1 and 2, series B, was higher on the last day than the first day of the run. This may be explained by the fact of the formation of the porous layer of oxides of arsenic and antimony on the anode which, at the time the voltage was read, did not drop off, and therefore offered resistance. In series A the anodes were scraped occasionally and readings were taken just after scraping. The diminution of potential difference may be due to the formation of nodular crystals, reducing the actual distance between the electrodes.

Experiment III.

In this experiment the cathodes were surrounded by diaphragms, the object being to prevent particles of impurity from depositing mechanically on the cathode. The diaphragm-frame was made of glass rod and was of semi-cylindrical shape (*d*, Fig. 1). The circular side and the bottom of the frame were closed with thin rubber dam, used by dentists, and the side which is between the electrodes with linen cloth. The cloth, before being used, was thoroughly washed with hot water, and the rubber dam was first treated with dilute sulphuric acid, then with water, in order to free it of any impurity that might have been present.

During the electrolysis one difficulty was encountered when the cathodes were surrounded with diaphragms. The latter, when soaked with electrolyte and in contact with the former, became cathodes, and thus copper was deposited on their sides and bottom, which soon bridged the electrodes and short-circuited the electric current. Moreover, rubber dam was found not a proper material because it deteriorated, became tender and increased the potential difference between the electrodes to a small extent.

The electrolysis was conducted at 60° C. and 30° C., and the electrolytes used for both series contained 2 percent and 6 percent arsenic, with the same proportion of cupric sulphate (15 percent) and free sulphuric acid (10 percent) as before.

As has already been stated, a sticky layer of oxides formed on the surface of the anode in the low-temperature series, whose formation caused the potential difference between the electrodes to be unusually high. To ascertain whether or not this high potential difference had any effect on the deposition of arsenic

and antimony on the cathode, the surface of two anodes (Nos. 1 and 3, series A, Table III) was occasionally scraped with a rubber "policeman" attached to a glass rod, while the surface of the other two anodes of series A (Nos. 2 and 4, Table III) was not disturbed.

Table III gives the results of this experiment and shows the averaged potential difference was much higher when the anode surface was not scraped than when scraped. The amount of arsenic in the deposited copper is practically the same in both cases, while the amount of antimony appears little higher in the case where the anode surface was not scraped.

TABLE III.

Time of experiment, 61 hours. Distance between electrodes, 1.75 inch (4.5 cm.). Current density, 40 amp. per sq. foot (4.4 amp. per sq. dm.). Electrolyte contained 15 percent $CuSO_4.5H_2O$ and 10 percent H_2SO_4. Anode contained 1.15 percent As and 1.05 percent Sb.

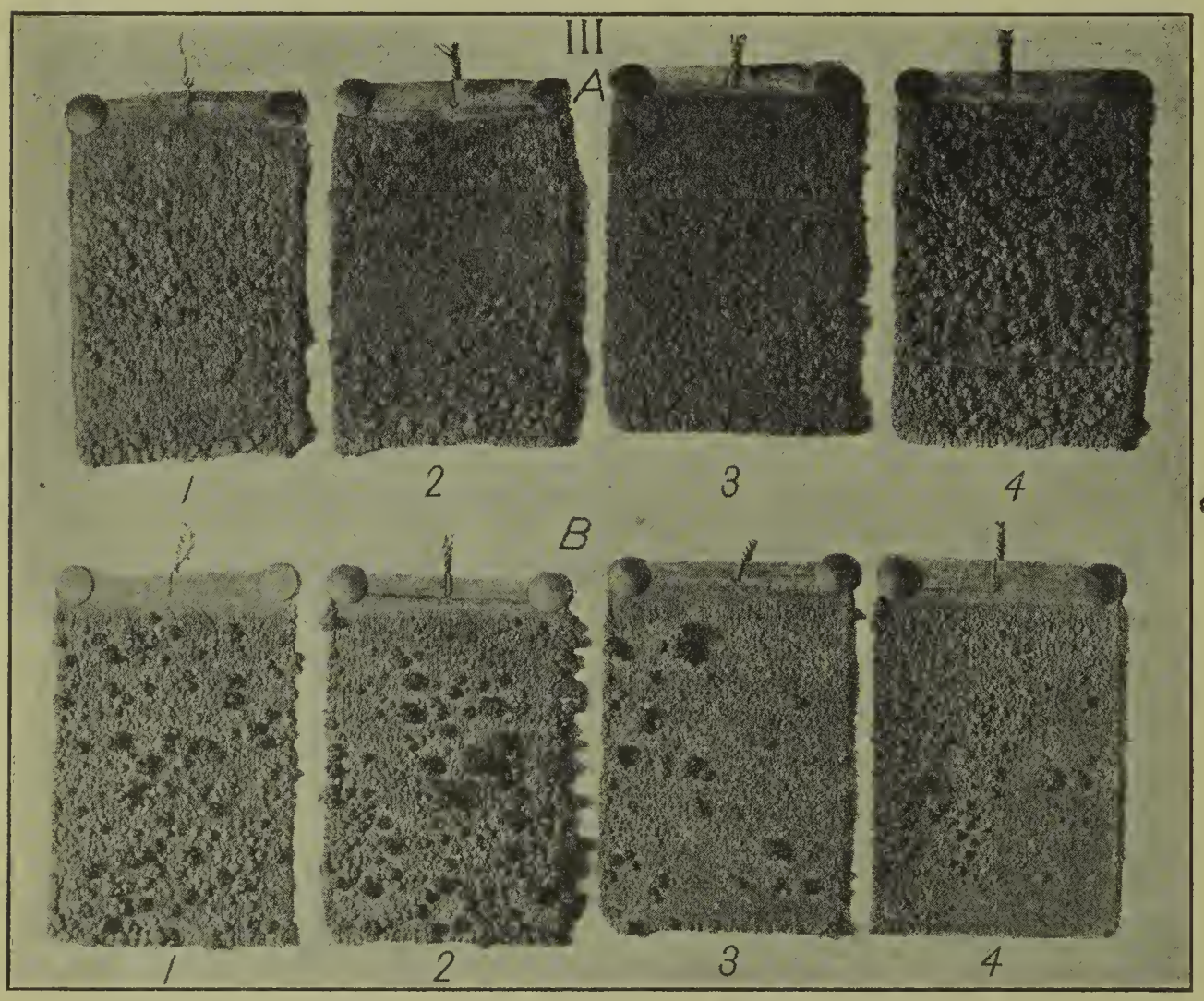
Number of Cells	Percent of As in Electrolyte	Temp. of Electrolyte in Degrees Cent.	Average E. M. F. in Volt	Impurities in Deposited Copper		Impurities in Elect. after Run		Photo III
				Percent As	Percent Sb.	Percent As	Percent Sb.	
Series A								A
1	2	30	0.57	0.118	0.099	2.40	0.003	1
2	2	30	0.79	0.115	0.129	2.11	None	2
3	6	30	0.62	0.124	0.053	5.80	0.003	3
4	6	30	1.12	0.160	0.079	6.10	0.002	4
Series B								B
1	2	60	0.49	0.186	0.268	2.28	0.004	1
2	2	60	0.44	0.171	0.222	2.21	0.002	2
3	6	60	0.44	0.025	0.001	6.28	0.002	3
4	6	60	0.46	0.031	0.001	6.30	0.004	4

As to the physical properties, the deposits which formed at 30° C. were all bad, brittle and high in arsenic and antimony, as is shown in Table III, and composed of coarse, nodular and incoherent crystals. The deposits which formed at 60° C. and in the electrolyte containing 2 percent arsenic were very bad. They were dark and became darker when exposed to the air. The analysis shows that they contained much arsenic and antimony. Those, on the other hand, which formed at the same temperature but in the electrolyte containing 6 percent arsenic, were good, bright and dense, though a few nodular crystals formed on the

surface and at the edges of the deposits. Photograph III, B3 and B4, shows their character.

Experiment IV.

In this experiment and in the rest of the experiments muslin diaphragms surrounded the anodes instead of the cathodes. These diaphragms were found satisfactory; short-circuiting was prevented and particles of impurities were col-



lected and, thus, prevented from collecting mechanically on the cathode.

The electrolytes used were prepared to contain 1.5 percent, 3 percent, 6 percent and 8 percent arsenic, and the amount of cupric sulphate and free sulphuric acid was 15 percent and 10 percent, respectively. The temperature of the electrolytes was 40° C. and 50° C. and the time of electrolysis was one hundred and four hours. Table IV gives the results of this experiment.

The deposits, No. 1, No. 2 and No. 3, series A, were all bad, rough, brittle and crystalline. Of these deposits, No. 2 and No. 3

were worse; they were composed of crystals easily detached, and consisted of long dendritic "trees" which interfered with the operation of stirrers and tended to short-circuit the current. Their surfaces were dull and became dark on exposure to the atmosphere. The chemical analyses show that they contain much arsenic and antimony. Deposit No. 4, series A, was good, solid, bright, and free from "trees," but consisted of a few small nodular crystals which scattered over the surface. It contained a very small amount of arsenic and antimony.

TABLE IV.

Time of experiment, 104 hours. Distance between electrodes, 1.75 inch (4.5 cm.). Current density, 40 amp. per sq. foot (4.4 amp. per sq. dm.). Electrolyte contained 15 percent $CuSO_4.5H_2O$ and 10 percent H_2SO_4. Anode contained 0.75 percent As and 0.73 percent Sb.

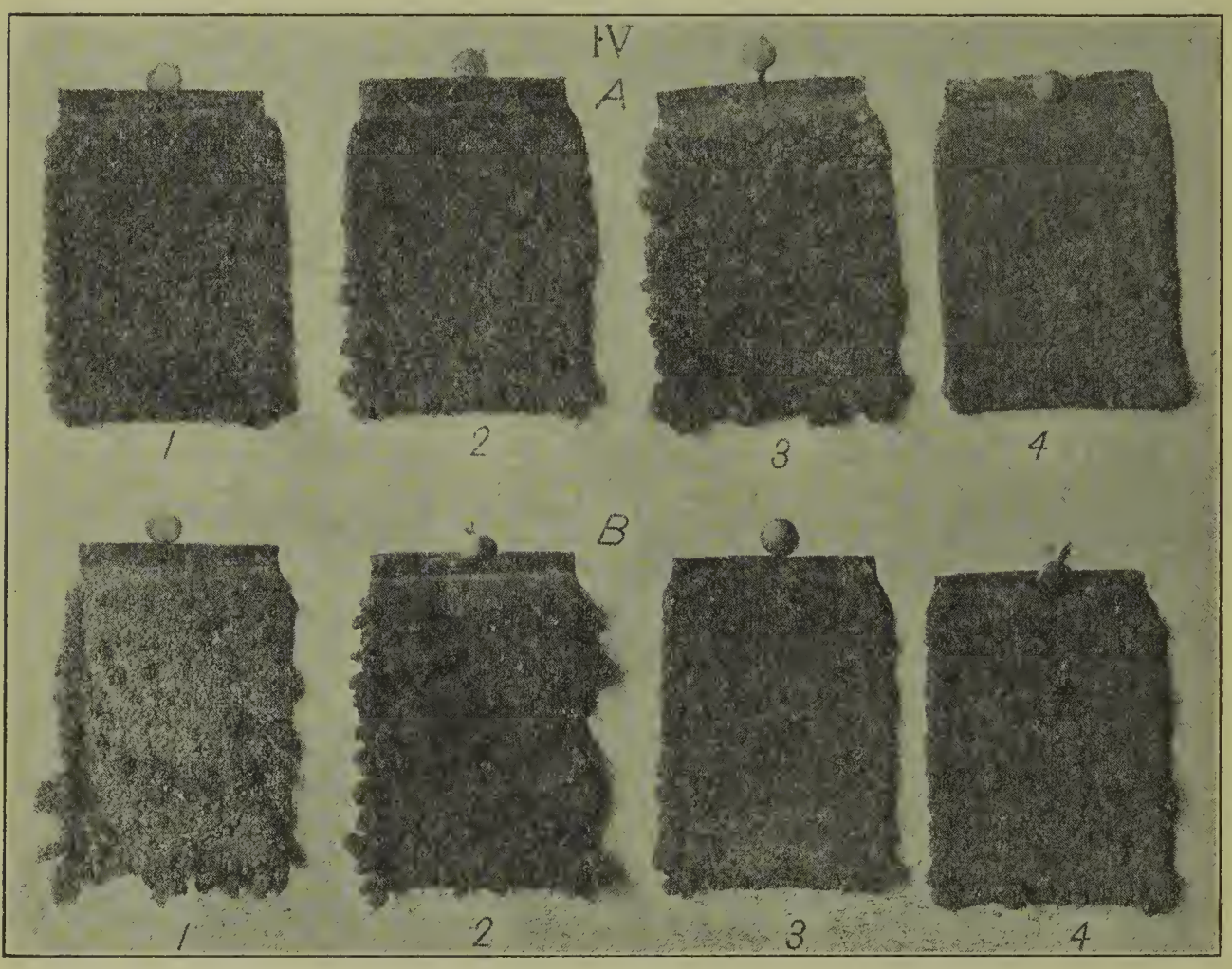
Number of Cells	Percent of As in Electrolyte	Temp. of Electrolyte in Degrees Cent.	Average E. M. F. in Volt	Impurities in Deposited Copper		Impurities in Elect. after Run		Photo IV
				Percent As	Percent Sb.	Percent As	Percent Sb.	
Series A								A
1	1.5	40	0.44	0.077	0.085	1.72	0.004	1
2	3.0	40	0.44	0.100	0.074	3.09	0.004	2
3	6.0	40	0.43	0.101	0.117	6.03	0.003	3
4	8.0	40	0.49	0.024	0.005	8.10	0.005	4
Series B								B
1	1.5	50	0.40	0.063	0.175	1.65	nil.	1
2	3.0	50	0.40	0.296	0.460	3.27	0.004	2
3	6.0	50	0.41	0.007	0.001	6.10	0.004	3
4	8.0	50	0.44	0.008	0.002	8.04	0.003	4

The deposits, No. 1 and No. 2, series B, which formed at 50° C. and in electrolytes containing 1.5 percent and 3 percent arsenic respectively, were also bad and brittle, and both were composed of long dendritic "trees." It was observed that deposit No. 2 was much worse than No. 1, as it was very brittle and dark. The analysis shows that both deposits contained high percentages of arsenic and antimony, but the percentage of these two impurities contained in deposit No. 2 was by far higher than that in deposit No. 1. As to the deposits No. 3 and No. 4, series B, which formed at the same temperature but in electrolytes containing 6 percent and 8 percent arsenic, they were found to be good, solid, bright and free from "trees," though a few rounded nodules

formed on the surface. They contained very low arsenic and antimony, as shown in Table IV. Photograph IV shows the character of the deposits.

Electrolytes Which Contained Inorganic "Addition-Agents."

This series of experiments was conducted with electrolytes of the same composition as those used in Experiment IV, with the



exception that an inorganic "addition-agent" was added. The temperature of electrolytes in series A was 40° C., and that in series B was 50° C.

Experiment V.

In this experiment sodium chloride was used as "addition-agent"; 0.1650 gram of this salt was weighed out and added to a liter of each electrolyte. In other words, the electrolytes contained 0.01 percent chlorine or 0.0065 percent sodium in the form of sodium chloride.

It may be pointed out here that when sodium chloride was added to the electrolyte it reacted with the cupric sulphate to form cupric chloride and sodium sulphate, as may be shown by the following equation:

$$(x + 2)NaCl + CuSO_4 = CuCl_2 + Na_2SO_4 + xNaCl.$$

Thus there were present in the electrolyte, in fact, three "addition-agents," instead of one, when sodium chloride was added.

The results of this experiment were very satisfactory and are shown in Table V.

TABLE V.

Time of experiment, 104 hours. Distance between electrodes, 1.75 inch (4.5 cm.). Current density, 40 amp. per sq. foot (4.4 amp. per sq. dm.). Electrolytes contained 15 percent $CuSO_4.5H_2O$ and 10 percent H_2SO_4 and 0.01 percent Cl as NaCl. Anode contained 0.87 percent As and 1.13 percent Sb.

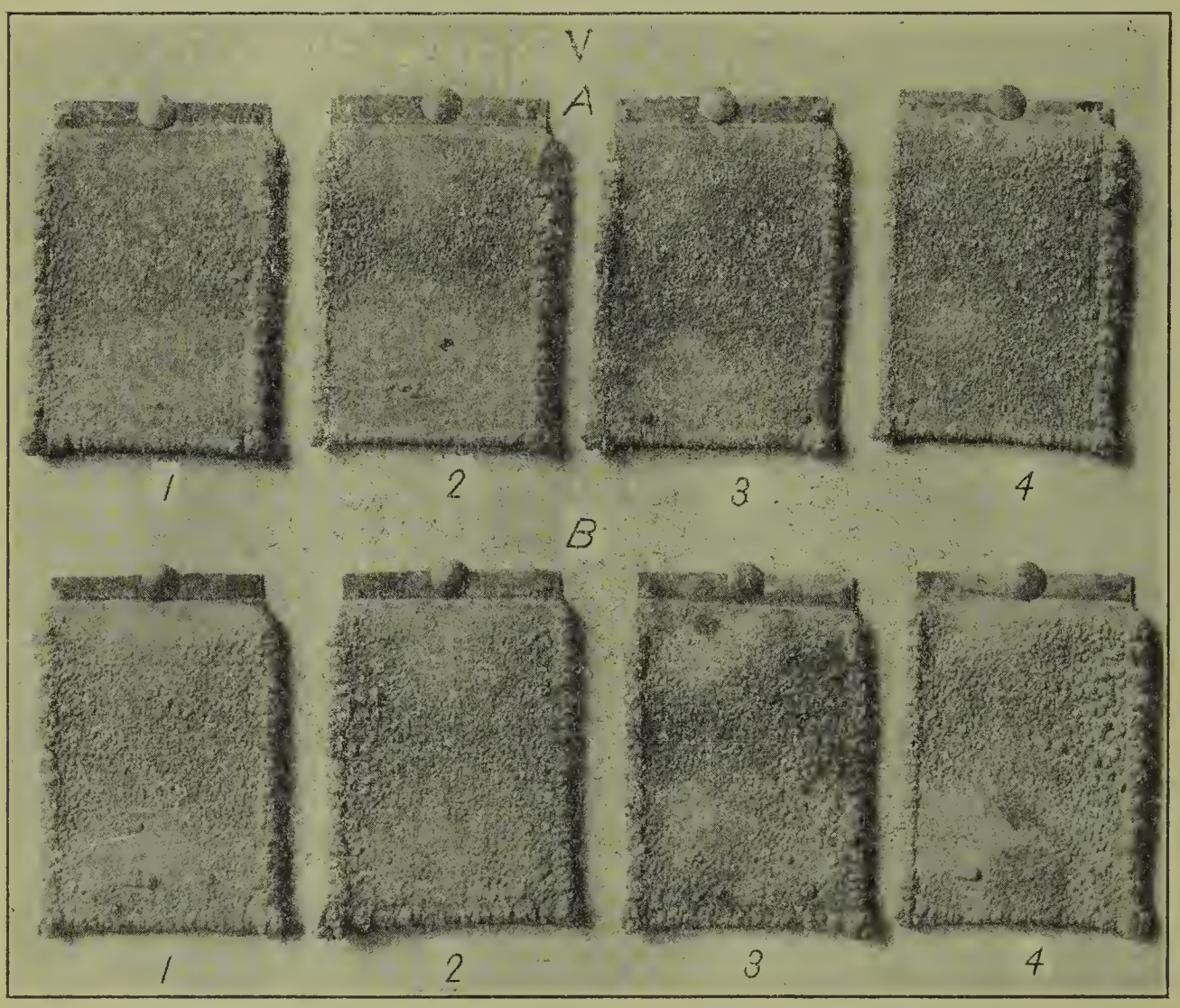
Number of Cells	Percent of As in Electrolyte	Temp. of Electrolyte in Degrees Cent.	Average E. M. F. in Volt	Impurities in Deposited Copper		Impurities in Elect. after Run		Photo V
				Percent As	Percent Sb.	Percent As	Percent Sb.	
Series A								A
1	1.5	40	0.48	0.0012	0.0005	1.97	0.0056	1
2	3.0	40	0.56	0.0006	0.0005	3.39	0.0036	2
3	6.0	40	0.51	0.0019	0.0008	6.59	0.0056	3
4	8.0	40	0.54	0.0019	0.0007	8.51	0.0098	4
Series B								B
1	1.5	50	0.47	0.0024	0.0003	1.90	0.0036	1
2	3.0	50	0.49	0.0012	0.0005	3.43	0.0097	2
3	6.0	50	0.47	0.0012	0.0005	6.70	0.0090	3
4	8.0	50	0.49	0.0006	0.0004	8.60	0.0058	4

The deposits from series A were very good, bright, solid, smooth, coherent and absolutely free from "trees." The deposits from series B were similar to those from A, except that their surfaces were a little brighter and the small crystals were a bit more pronounced. The analyses of these deposits show that the arsenic and antimony were very low in every case.

In addition to the chemical determination of the impurities in the cathode copper, a physical bending test was also made. A strip of the cathode copper, about ¾ inch wide, 3¾ inches long, was taken for this test. It was bent, with the under-side out, and hammered double; in no case did the strips crack at the

bend. This bending test showed that the cathode copper was very ductile and of high purity in all cases.

The addition of such a small amount of sodium chloride was found to exert a remarkably favorable influence upon the deposited copper, for it not only improved the physical properties of the deposits, but also overcame the formation of "sprouts" and prevented the precipitation of arsenic and antimony with the



cathode copper. (Compare results given in Tables IV and V.) Photograph V shows the character of the deposits.

Experiment VI.

As Experiment V shows that the presence of a small amount of sodium chloride in the electrolytes exerts a marked beneficial influence upon the copper deposits, it now becomes important to ascertain which of the two ions (Na ion, or Cl ion) produces this good effect. To attain this object, hydrochloric acid, which has the Cl ion in common with sodium chloride, was first tried and

used as "addition-agent" in this experiment. For this purpose, standard hydrochloric acid solution was prepared by diluting 10 c.c. of concentrated acid (containing 37.5 percent HCl) to 100 c.c. A measured volume of this solution was added to each electrolyte so that it contained the same amount of chlorine, per liter, as in the case of sodium chloride, that is, 0.01 percent chlorine.

It may be noted here that just as sodium chloride reacts with the cupric sulphate of the electrolyte, so hydrochloric acid reacts with it, to form cupric chloride. This reaction may be represented by the following equation:

$$(x + 2)HCl + CuSO_4 = H_2SO_4 + CuCl_2 + xHCl.$$

So, in reality, there also exists in the electrolyte cupric chloride as well as hydrochloric acid, when the latter is added to the cupric sulphate electrolyte. The experimental data are given in Table VI.

TABLE VI.

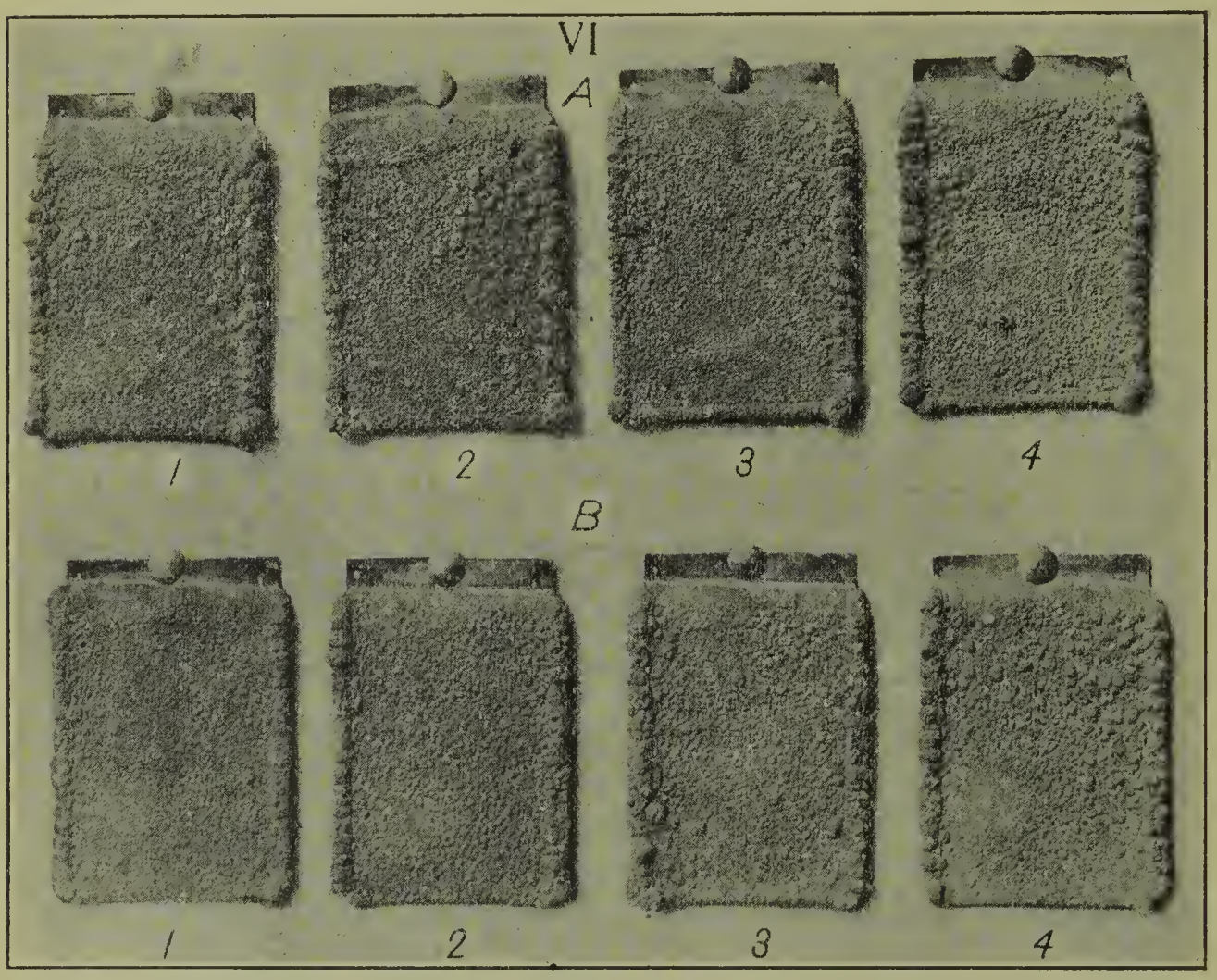
Time of experiment, 104 hours. Distance between electrodes, 1.75 inch (4.5 cm.). Current density, 40 amp. per sq. foot (4.4 amp. per sq. dm). Electrolyte contained 15 percent $CuSO_4.5H_2O$, 10 percent H_2SO_4, and 0.01 percent Cl as HCl. Anode contained 1.15 percent As and 1.12 percent Sb.

Number of Cells	Percent of As in Electrolyte	Temp. of Electrolyte in Degrees Cent.	Average E. M. F. in Volt	Impurities in Deposited Copper		Impurities in Elect. after Run		Photo VI
				Percent As	Percent Sb.	Percent As	Percent Sb.	
Series A								A
1	1.5	40	0.49	0.0022	0.0004	1.83	0.0070	1
2	3.0	40	0.47	0.0030	0.0004	3.24	0.0036	2
3	6.0	40	0.48	0.0074	0.0005	6.04	0.0080	3
4	8.0	40	0.49	0.0106	0.0004	8.06	0.0030	4
Series B								B
1	1.5	50	0.45	0.0015	0.0009	2.09	0.0075	1
2	3.0	50	0.44	0.0014	0.0002	3.46	0.0083	2
2	6.0	50	0.44	0.0037	0.0007	6.25	0.0030	3
4	8.0	50	0.44	0.0023	0.0007	8.31	0.0030	4

The deposits formed at both temperatures (40° C. and 50° C.) were satisfactory. Those which formed at 40° C. were bright, solid, smooth, slightly crystalline deposits, free from "trees," but nodular at their edges, whereas the deposits formed at 50° C. were similar in character, but brighter. As to the result of bend-

ing test, the deposits produced at the higher temperature appeared to be more ductile than those formed at the lower temperature, as they cracked less than those formed at 40° C. When the test-strips of deposits No. 3 and No. 4, series A, were hammered double, they cracked at the outer side of the bend.

Chemical analyses show that the deposits formed at 40° C. were higher in arsenic than those at 50° C. and that they were



not so pure as those obtained in electrolytes containing sodium chloride.

The results of this experiment, therefore, indicate that hydrochloric acid is not so active an addition-agent as sodium chloride, as the presence of the latter gives cathode copper which is purer and more ductile. Photograph VI shows the character of the deposits.

Experiment VII.

As Cl ion was found to produce a good effect upon the deposited copper and to prevent, to a great extent, the deposition

of arsenic and antimony, it now remained to find whether the Na ion would produce the same effect. In order to accomplish this, sodium sulphate ($Na_2SO_4.10H_2O$) was selected and a weighed amount of this salt added to each electrolyte, so that it contained 0.0065 percent sodium, which corresponded to the same amount of sodium as in the case of sodium chloride (Experiment V).

Table VII gives the results of this experiment and Photograph VII shows the character of the deposits.

TABLE VII.

Time of experiment, 104 hours. Distance between electrodes, 1.75 inch (4.5 cm.). Current density, 40 amp. per sq. foot (4.4 amp. per sq. dm.). Electrolyte contained 15 percent $CuSO_4.5H_2O$, 10 percent H_2SO_4, and 0.0065 percent Na as Na_2SO_4. Anode contained 1.47 percent As and 1.12 percent Sb.

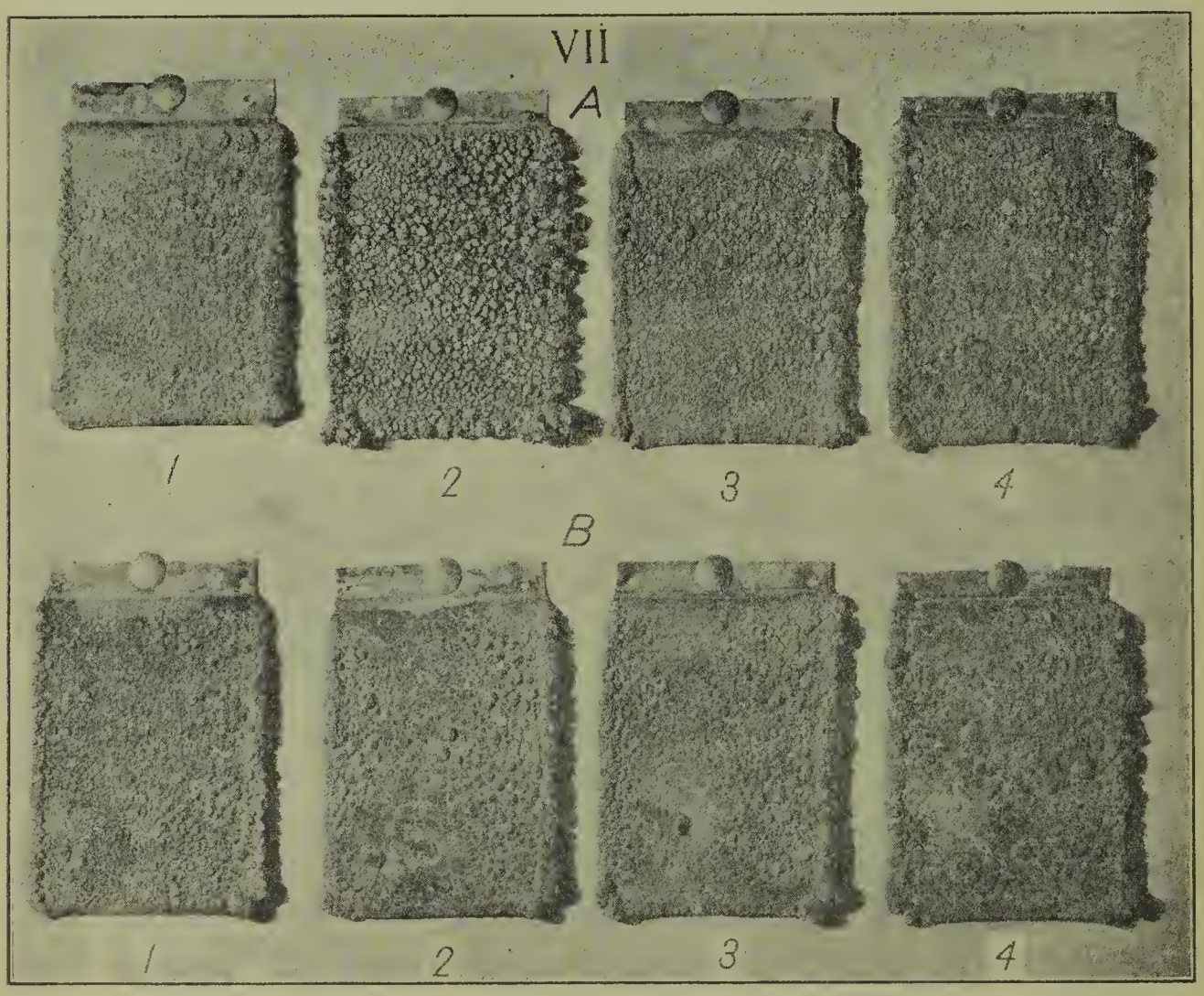
Number of Cells	Percent of As in Electrolyte	Temp. of Electrolyte in Degrees Cent.	Average E. M. F. in Volt	Impurities in Deposited Copper		Impurities in Elect. after Run		Photo VII
				Percent As	Percent Sb.	Percent As	Percent Sb.	
Series A								A
1	1.5	40	0.51	0.0023	0.0006	1.86	0.0017	1
2	3.0	40	0.50	0.0975	0.1403	3.36	0.0090	2
3	6.0	40	0.51	0.0066	0.0016	6.16	0.0084	3
4	8.0	40	0.50	0.0061	0.0004	8.44	0.0060	4
Series B								B
1	1.5	50	0.45	0.0011	0.0005	1.97	0.0045	1
2	3.0	50	0.46	0.0023	0.0003	3.32	0.0021	2
3	6.0	50	0.44	0.0018	0.0002	6.37	0.0073	3
4	8.0	50	0.46	0.0063	0.0004	8.45	0.0056	4

The deposits which formed at 40° C. and in electrolytes containing 1.5 percent, 6 percent and 8 percent arsenic were bright, solid and free from "trees," while the deposit which formed at the same temperature but in the electrolyte which contained 3 percent arsenic was very bad, exceedingly brittle and of dull color. It was composed not only of nodular crystals, but of dendritic "trees" formed especially at the edges, as clearly shown in No. 2, Photograph VII—*A*.

When the test-strips of deposits of series A were hammered double it was found that they were not very ductile, as deposits No. 3 and No. 4 were broken into two, and deposit No. 1 almost into two.

The deposits which formed at 50° C. were all bright and solid, and free from "trees," though a few small rounded nodules formed on the surface. The bending test showed that these deposits were much tougher and more ductile than those which formed at 40° C., for the test-strips did not even crack when they were hammered double.

As to the purity of the deposited copper, it will be observed in Table VII that the amount of arsenic and antimony in the



cathodes of both series was in all cases low, except deposit No. 2, series A, which was formed at 40° C. and in electrolyte containing 3 percent arsenic. It contained much arsenic and antimony. It should also be observed that the amount of impurities contained in deposits which formed at 40° C. was notably higher than that contained in those which formed at 50° C.

From what has been said, it will then be seen that the presence of a *small* amount of sodium sulphate in the electrolytes has a decidedly good effect, chemically and physically, upon the de-

posited copper, provided that the electrolysis be conducted at 50° C. At 40° C., however, its effect was somewhat decreased and it had practically no effect upon the deposited copper when the electrolyte contained 3 percent arsenic.

As to the relative effectiveness of the Na ion and the Cl ion, the former appeared to be less effective at the lower temperatures, whereas at the higher temperature the beneficial effect of the presence of either of the ions was appreciably the same.

Experiment VIII.

In this experiment, a larger amount of sodium sulphate was employed than in Experiment VII. It was added to the electrolytes with the object of ascertaining whether the larger amount would produce a better effect upon the deposited copper. The amount added was 100 times that which was used in Experiment VII; in other words, each of the electrolytes contained 0.65 percent sodium as sodium sulphate.

TABLE VIII.

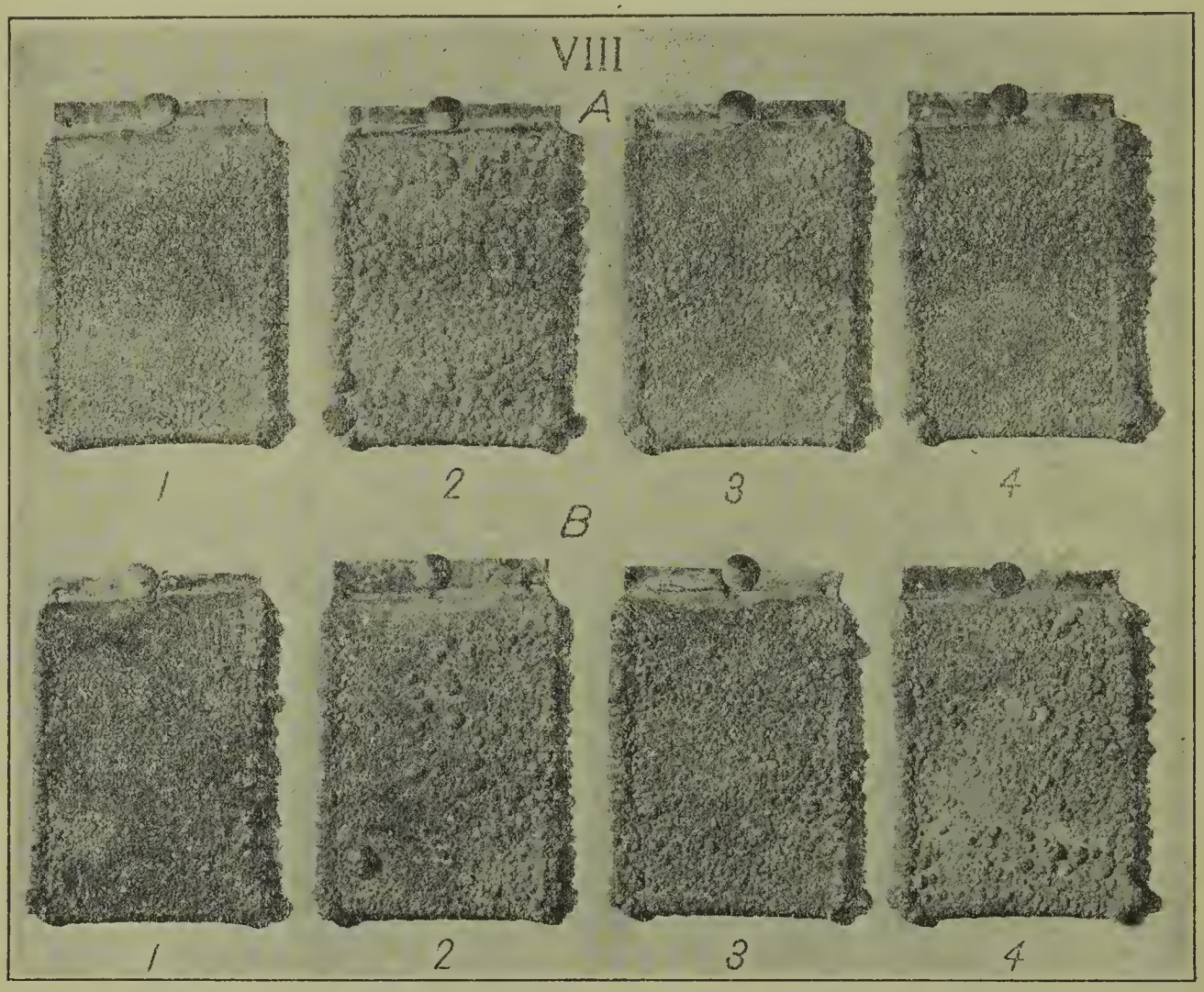
Time of experiment, 104 hours. Distance between electrodes, 1.75 inch (4.5 cm.). Current density, 40 amp. per sq. foot (4.4 amp. per sq. dm.). Electrolyte contained 15 percent $CuSO_4.5H_2O$, 10 percent H_2SO_4, and 0.65 percent Na as Na_2SO_4. Anode contained 0.98 percent As and 0.75 percent Sb.

Number of Cells	Percent of As in Electrolyte	Temp. of Electrolyte in Degrees Cent.	Average E. M. F. in Volt	Impurities in Deposited Copper		Impurities in Elect. after Run		Photo VIII
				Percent As	Percent Sb.	Percent As	Percent Sb.	
Series A								A
1	1.5	40	0.54	0.0020	0.0002	1.85	0.0040	1
2	3.0	40	0.52	0.0051	0.0028	3.16	0.0021	2
3	6.0	40	0.51	0.0086	0.0006	6.02	0.0016	3
4	8.0	40	0.54	0.0104	0.0002	8.20	0.0029	4
Series B								B
1	1.5	50	0.44	0.0013	0.0004	1.85	0.0015	1
2	3.0	50	0.44	0.0018	0.0004	3.42	0.0058	2
3	6.0	50	0.45	0.0019	0.0006	6.34	0.0054	3
4	8.0	50	0.45	0.0023	0.0007	8.05	0.0032	4

It should be mentioned here that during the electrolysis the potential difference, particularly in the lower temperature series, seemed to increase slightly with the presence of this large amount of sodium sulphate.

The deposits which formed at both temperatures were satisfactory. They were bright, smooth, tough and solid, though a few small rounded nodules were scattered on the surface. Deposit No. 2, series A, was the most nodular. The bending test showed that they were ductile, and that those formed at the higher temperature were more ductile than those formed at the lower temperature.

The chemical analyses show that the deposits formed at 40° C.



contained higher percentage of arsenic than those which formed at 50° C., while the percentages of antimony in both series were low, except No. 2, series A, which contained a much greater percentage than the rest. The experimental data of this experiment are given in Table VIII and the character of the deposits is shown in Photograph VIII.

Comparing the results with those obtained in Experiment VII, it is found that the larger amount of sodium sulphate added to the electrolytes does not appear to have much improved the copper

deposits of either series, except the deposit which formed at 40° C. and in the electrolyte containing 3 percent arsenic, in which case it hindered to a great extent the precipitation of arsenic and antimony.

It is therefore obvious from the results of the two experiments (VII and VIII) that at 50° C. the presence of a small amount of sodium sulphate had a decidedly good effect on the deposited copper in all cases, whereas at 40° C. such a small amount had the same effect, but no effect in the case in which the electrolyte contained 3 percent arsenic, unless a sufficiently large amount was present. The higher temperature is therefore preferable, as it not only causes the deposited copper to form more ductile, but also in this case renders the "addition-agent" more effective.

Experiment IX-A.

The electrodes used in this experiment contained only 3 percent and 6 percent arsenic, as the electrolysis was conducted only at 50° C. The "addition-agents" tried were aluminium sulphate ($Al_2(SO_4)_3.18H_2O$) and aluminium chloride ($AlCl_3.6H_2O$). In each case the amount of salt added was such that the electrolyte contained 0.10 percent Al.

TABLE IX—*A*.

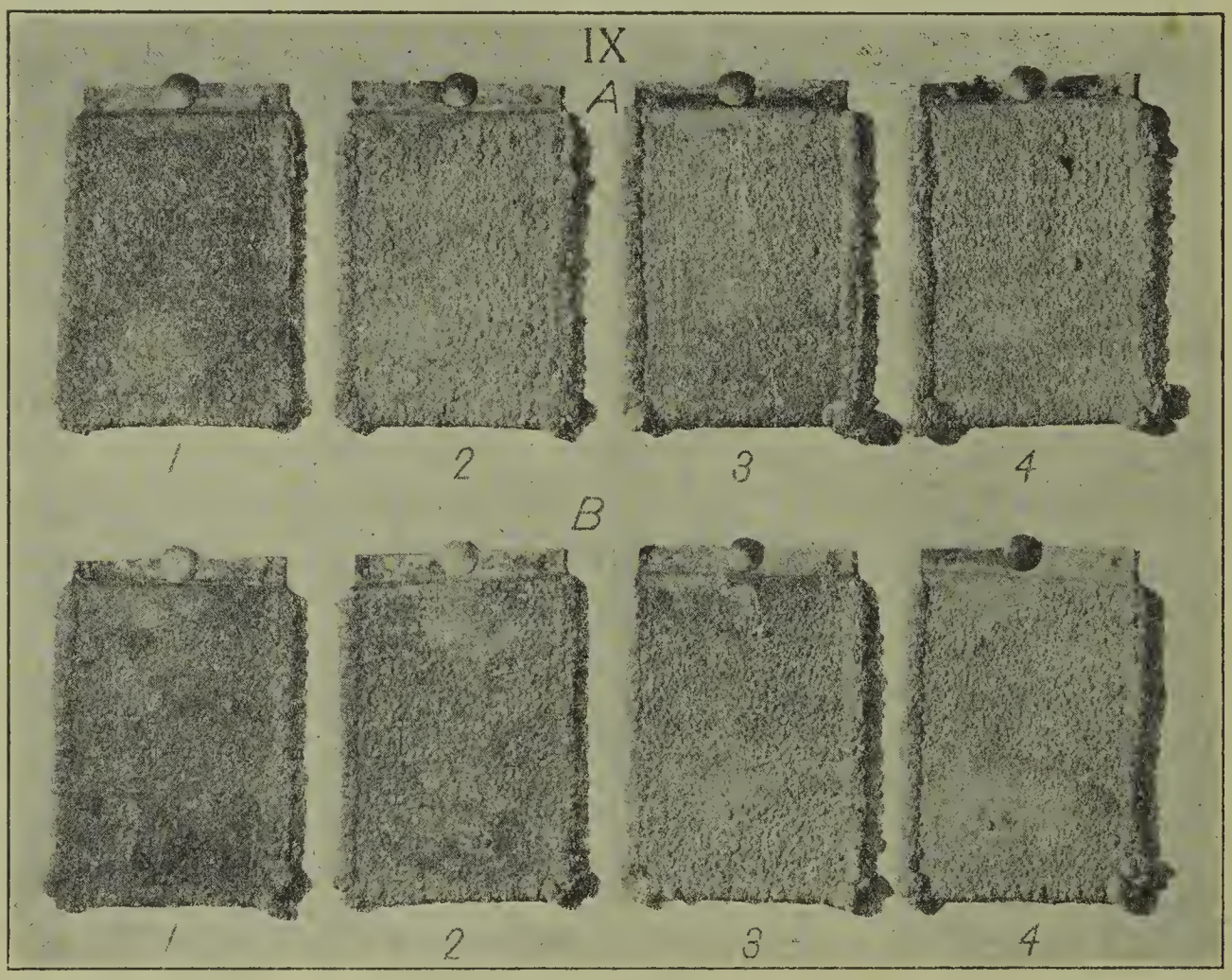
Time of experiment, 104 hours. Distance between electrodes, 1.75 inch (4.5 cm.). Current density, 40 amp. per sq. foot (4.4 amp. per sq. dm.). Electrolyte contained 15 percent $CuSO_4.5H_2O$ and 10 percent H_2SO_4. Temperature of electrolyte 50° C. Anode contained 0.97 percent As and 0.89 percent Sb.

No. of Cells	Percent of As in Electrolyte	Addition-Agent		Average E. M. F. in Volt	Impurities in Deposited Copper		Impurities in Elect. after Run		Photo IX-A
		$Al_2(SO_4)_3$ % in Al.	$AlCl_3$ % in Al.		Percent As	Percent Sb.	Percent As	Percent Sb.	
1	3.0	0.10	. .	0.47	0.0028	0.0003	3.44	0.0061	1
2	6.0	0.10	.	0.47	0.0050	0.0003	6.48	0.0095	2
*3	3.0	. .	0.10	0.45	0.0030	0.0004	3.08	0.0081	3
*4	6.0	. .	0.10	0.43	0.0087	0.0004	5.92	0.0041	4

It should be mentioned here that, at the beginning of the electrolysis, a peculiar effect was observed in the case in which alu-

*The bad copper first deposited was filed off before the samples were taken for analysis.

minium chloride was used. A white layer, which appeared to be oxide of arsenic, formed on the cathode surface as soon as electrolysis began and in the course of a few hours became darker. During the formation of this layer the potential difference was high, reading from 0.58 to 0.60 volt. After a period of fifteen hours, however, the deposited copper began to improve, giving a bright surface. This action of aluminium chloride is difficult to explain at present, unless it precipitated arsenic from solution at the first period of the run. A satisfactory explanation may be



had with further investigations. But suffice it to say that the strange effect might be due to the presence of an excessive amount of aluminium chloride in the electrolyte; these electrolytes, at the end of the run, contained less arsenic than those to which aluminium sulphate was added.

The two deposits from electrolytes containing aluminium chloride were found each to consist of two layers. The layer first deposited was bad, brittle and incoherent, while the other layer, on the contrary, was bright, smooth and quite coherent. The

analytical results of No. 3 and No. 4, Table IX—*A*, show that both of these layers were low in arsenic and antimony. The character of these two deposits is shown in Photograph IX—*A*, marked 3 and 4.

With aluminium sulphate in the electrolytes, no such action as that of aluminium chloride was observed. The presence of aluminium sulphate rendered the deposited copper bright, solid and coherent, and prevented the deposition of arsenic and antimony, as is clearly shown in Table IX—*A*, No. 1 and No. 2. The deposits also possessed great ductility, which the test-strips indicated when they were hammered double.

Experiment IX—B.

With a view to determining the relative effect of a larger or a smaller amount of sodium chloride upon copper deposits, this experiment was performed with electrolytes of the same composition as was used in Experiment IX—*A*, but with the addition of two different amounts of sodium chloride. In the one case the amount of salt added was such that the electrolyte contained 0.05 percent Cl, and in the other case the electrolyte was made to contain twice as much, that is to say, 0.10 percent Cl.

The deposits in both cases were similar in character, but those from electrolytes containing less sodium chloride appeared to be little better, particularly in color. All the deposits were bright, solid, smooth and free of nodules, but they, as compared with those obtained from electrolytes which contained only 0.01 percent Cl (Experiment V) seemed to be a little inferior in physical properties, that is, less bright, less smooth and less ductile.

The result of bending test showed that the deposits from electrolytes containing 0.05 percent Cl were more ductile than those which formed in electrolytes containing 0.10 percent, as the test-strips of the latter cracked almost in two, while those of the former did not crack when they were hammered double.

The purity of the deposits was in all cases high, but was not so high as those from electrolytes which contained 0.01 percent Cl as sodium chloride, when the electrolysis was conducted under similar conditions. The results of this experiment are expressed

in Table IX—*B*, and the character of the deposits are shown in Photograph IX—*B*.

TABLE IX—*B*.

Time of experiment, 104 hours. Distance between electrodes, 1.75 inch (4.5 cm.). Current density, 40 amp. per sq. foot (4.4 amp. per sq. dm.) Electrolyte contained 15 percent $CuSO_4.5H_2O$ and 10 percent H_2SO_4. Temperature of electrolyte 50° C. Anode contained 0.97 percent As and 0.89 percent Sb.

No. of Cells	Percent of As in Electrolyte	Addition Agent NaCl		Average E. M. F. in Volt	Impurities in Deposited Copper		Impurities in Elect. after Run		Photo IX-B
		Percent in Cl.	———		Percent As	Percent Sb.	Percent As	Percent Sb.	
1	3.0	0.10	. .	0.43	0.0022	0.0004	3.50	0.0078	1
2	6.0	0.10	. .	0.42	0.0048	0.0007	6.48	0.0064	2
3	3.0	0.05	. .	0.41	0.0020	0.0008	3.41	0.0223	3
4	6.0	0.05	. .	0.41	0.0018	0.0003	6.41	0.0190	4

From the results of this experiment and those of Experiment V, series B, it will be seen that the presence of a greater amount of sodium chloride in the electrolyte proved to be less beneficial on the deposited copper. Therefore, when sodium chloride is used to improve the character of the deposits and to prevent the deposition of arsenic and antimony, the amount added to the electrolyte had better be limited to not more than 0.01 percent Cl.

Electrolytes Which Contained Organic "Addition-Agents."

In this series of experiments, the organic "addition-agents" tried were gelatine, tannin, and peptone (E. & A. dry, from meat) and the electrolyses were conducted with electrolytes of the following composition:

(*a*) Electrolyte containing 15 percent $CuSO_4,5H_2O$, 10 percent H_2SO_4 plus 3 percent arsenic, and 0.01 percent organic addition-agent.

(*b*) Electrolyte containing 15 percent $CuSO_4.5H_2O$, 10 percent H_2SO_4 plus 6 percent arsenic and 0.01 percent organic addition-agent.

(*c*) Electrolyte containing 15 percent $CuSO_4.5H_2O$, 10 percent H_2SO_4 plus 3 percent arsenic and 0.02 percent organic addition-agent.

(*d*) Electrolyte containing 15 percent $CuSO_4.5H_2O$, 10 percent H_2SO_4 plus 6 percent arsenic and 0.02 percent organic addition-agent.

The gelatine and tannin were each prepared by dissolving 5 grams in 100 c.c. of boiling water. The solution was then diluted to 200 c.c., which contained 2.5 percent of the reagent. The solution of peptone was prepared in the same way, but dissolved in a larger quantity of water (about 350 c.c.) and diluted to 400 c.c., for peptone is not so soluble as the other two organic substances. This solution contained 1.25 percent peptone.

Experiment X.

In this experiment gelatine and tannin were used as "addition-agents," and the amount added in each case was such that the electrolytes contained one part, by weight, of the "addition-agent" to 10,000 parts of electrolyte, and one part to 5,000 parts, that is to say, the electrolytes contained 0.01 percent and 0.02 percent of the organic "addition-agent."

With the presence of gelatine in the electrolyte, it was observed that during the first few hours of electrolysis, the gelatine produced a strange effect upon the deposited copper, as small "trees," which are shown in C, Photograph X, were formed in every case all over the cathode surface and particularly at the edges. The higher the proportion of gelatine in the electrolytes, the more and larger the "trees," some of which grew as long as half an inch at the end of fifteen hours' electrolysis. At this period the cathodes were withdrawn and the "trees" knocked off. The cathodes were again placed in the original position, but with the reverse sides front, in order to ascertain whether the gelatine had the same effect after fifteen hours electrolysis. It was, however, surprising to find that the copper which deposited from this time on to be perfectly smooth, and that neither "trees" nor nodules were formed on the cathode surface.

Another fact that was also observed during the electrolysis was

that the presence of gelatine caused the potential difference to be much higher, an average of about 0.04 to 0.08 volt; and, as the electrolysis continued, it gradually diminished.

From the two facts mentioned above, it appears that gelatine, as the result of electrolysis, undergoes a chemical change and the product or products thus formed must have produced such a beneficial influence as not only to prevent the formation of "trees" and the deposition of arsenic and antimony, but also to cause the deposited copper to form smooth and tough.

TABLE X.

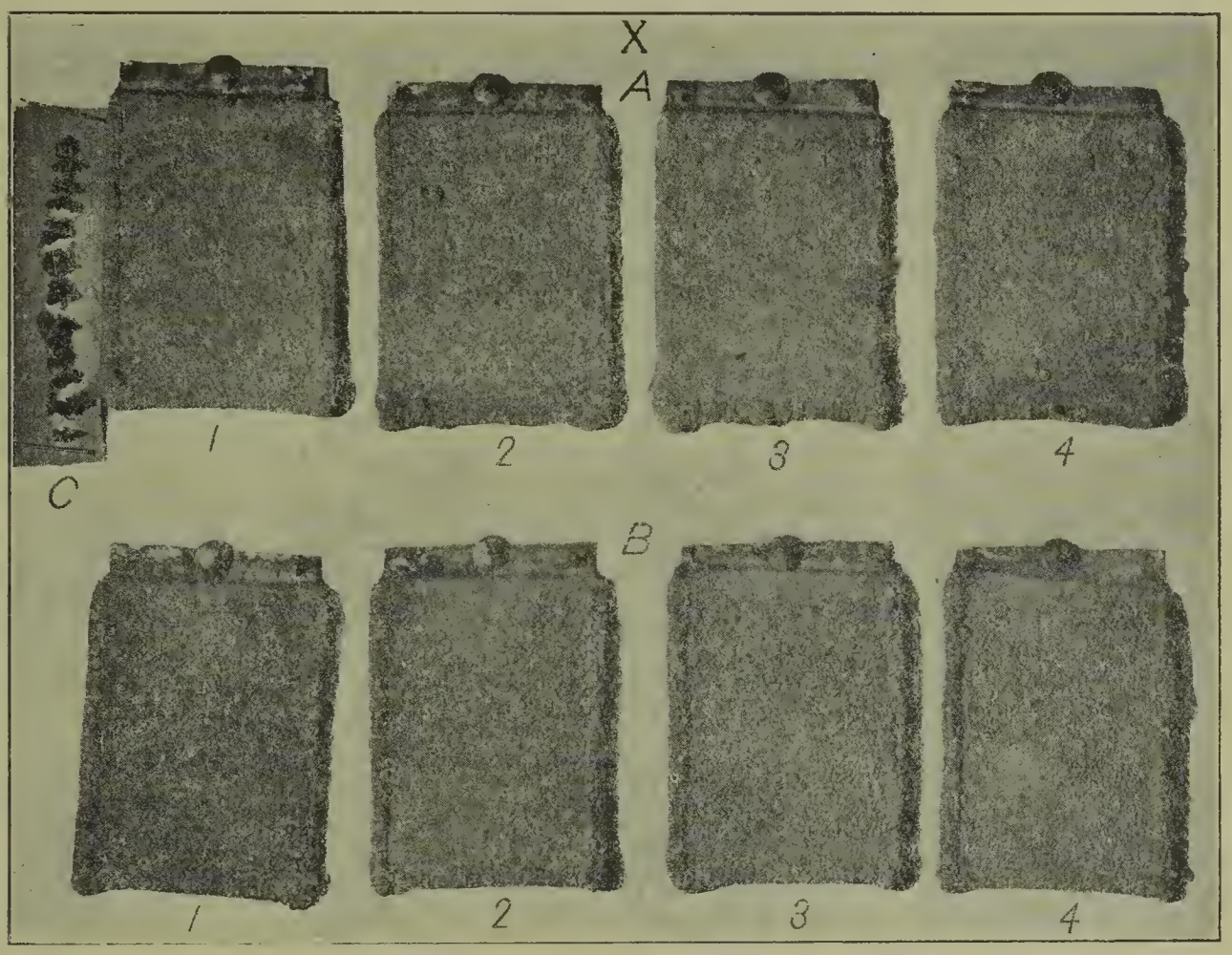
Time of experiment, 104 hours. Distance between electrodes, 1.75 inch (4.5 cm.). Current density, 40 amp. per sq. foot (4.4 amp. per sq. dm.). Electrolyte contained 15 percent $CuSO_4.5H_2O$ and 10 percent H_2SO_4. Temperature of Electrolyte 50° C. Anode contained 1.06 percent As and 1.01 percent Sb.

Number of Cells	Percent of As in Electrolyte	Addition-Agents		Average E. M. F. in Volt	Impurities in Deposited Copper		Impurities in Elect. after Run		Photo X
		Percent Gelatine	Percent Tannin		Percent As	Percent Sb.	Percent As	Percent Sb.	
Series A									A
1	3.0	0.01	. .	0.48	0.0013	0.0003	3.19	0.0023	1
2	6.0	0.01	. .	0.48	0.0018	0.0003	6.11	0.0023	2
3	3.0	0.02	. .	0.48	0.0009	0.0003	3.17	0.0019	3
4	6.0	0.02	. .	0.50	0.0020	0.0007	5.97	0.0062	4
Series B									B
1	3.0	. .	0.01	0.46	0.0020	0.0002	3.12	0.0023	1
2	6.0	. .	0.01	0.45	0.0015	0.0003	6.18	0.0033	2
3	3.0	. .	0.02	0.41	0.0012	0.0002	3.25	0.0039	3
4	6.0	. .	0.02	0.45	0.0017	0.0002	6.16	0.0042	4

With tannin in the electrolyte, the copper deposited was observed to be perfectly smooth throughout the run, and after fifteen hours electrolysis the cathodes were turned reverse side front, in the same manner as in the case of those in the electrolytes containing gelatine. This was done in order to show whether tannin would produce different effect on the deposited copper after a period of electrolysis. The presence of tannin in the electrolytes did not raise the potential difference to any extent and it was in every case lower than the electrolytes which contained gelatine.

After forty-two hours run the deposited copper formed in electrolytes containing tannin, in each case, was observed to become spangling, and the crystals appeared to have sharp edges, which might be due to the "addition-agent" having been consumed. At this period a further addition of tannin was therefore made, and the amount added to each cell was the same as originally present. But this addition did not cause any apparent improvement of the deposits.

The results of this experiment are recorded in Table X.



The deposits, series A, which formed in electrolytes containing gelatine, were very bright and solid, perfectly smooth and coherent, and composed of very fine grains. They also possessed very high ductility, as the test-strips did not crack nor show even signs of cracking when doubled and hammered flat.

The physical and mechanical properties of the deposits, series B, which formed in the presence of tannin, were similar to those of series A, except that the grains of the deposits were coarse and more pronounced. They possessed very high ductility, similar to

deposits from electrolytes which contained gelatine. Photograph X shows the character of the deposits of these two series.

The purity of the deposited copper from both series was, in every case, extraordinarily high. The analyses show that they contained in no case more than 0.002 percent arsenic and 0.0007 percent antimony.

From what has been said regarding the physical and chemical properties of the deposits obtained from both series, it will be seen that the presence of gelatine in the electrolytes, though at the start it caused the formation of small "trees," produced a remarkably beneficial effect upon the deposited copper, and that the effect of tannin was very much the same. Both of these organic "addition-agents" rendered the deposits smooth, very ductile and finely crystalline, and were very effective in preventing the deposition of arsenic and antimony. Of these two organic substances, tannin seemed to be preferable with such conditions as were maintained during this experiment; no "trees" formed at the start of the electrolysis.

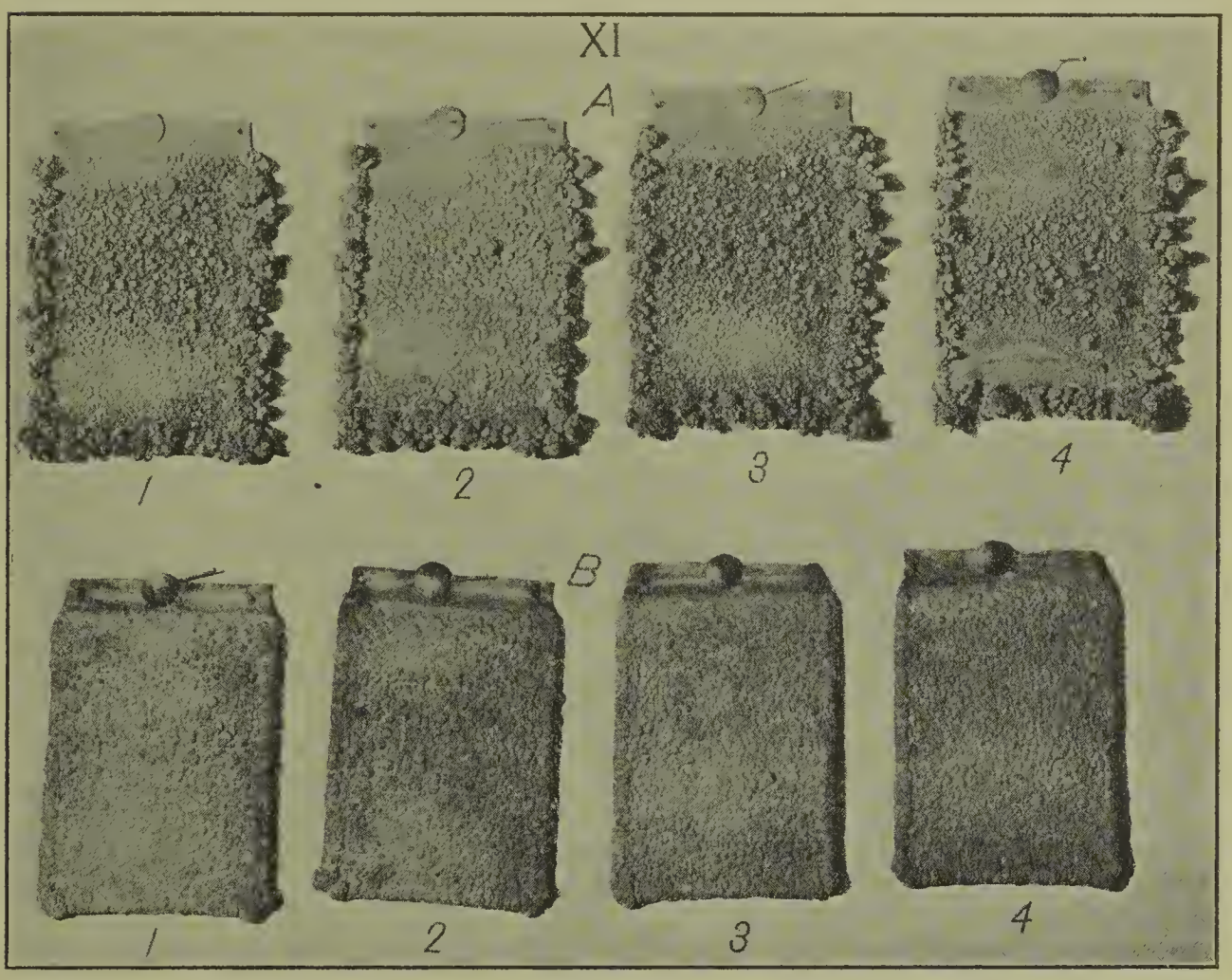
Experiment XI-A.

Peptone (dry, from meat) was employed in this experiment as "addition-agent"; the amount added at the start to the electrolytes was in the same proportion as gelatine and tannin in the preceding experiment, that is, one part, by weight, of peptone in 10,000 parts, and 5,000 parts of each electrolyte.

It may be stated here that the presence of peptone produced a strange and bad effect on the deposited copper. Small "trees" formed at the beginning of the run, which looked like those formed at the start in electrolytes containing gelatine, but of duller color. After an interval of fifteen hours from the beginning of the electrolysis, the cathodes were withdrawn and, after the "trees" at the edges were knocked off, again placed in the original position, but with the reverse sides front, in order to ascertain whether the peptone, as in the case of gelatine, would then behave differently. A further addition of the peptone was made at this time—same amount as originally present in each cell. During the three hours after this addition, the surface of the cathodes seemed to form smooth and spangling, but soon afterwards the same kind of "trees" began to appear, continued to

grow, and became large and long at the end of the run, as shown in Photograph XI-*A*.

Another fact that was observed during the electrolyses is that the potential differences between the electrodes in each case dropped greatly, but gradually, and whenever an addition of peptone was made the potential difference was increased from 0.06 to 0.08 volt. This shows that the drop of voltage was an indication of the consumption of the organic "addition-agent." In order, then, to maintain a certain amount of peptone present



in the electrolytes, it became necessary to add a certain amount at such intervals as the drop in voltage was observed. Two further additions were made, and the amount added each time to the cells was in the proportion of one part, by weight, of "addition-agent" to 5,000 parts of electrolyte.

The results of this experiment are recorded in Table XI-*A*, and the character of the deposits are shown in Photograph XI-*A*.

None of the deposits from this experiment were at all satisfactory. They were exceedingly brittle and their color was dark;

they were also composed of coarse, loosely adherent crystals and of dendritic "trees" easily detached. The latter interfered with the electrolysis and greatly decreased the potential between the electrodes, thus causing the average potential difference to become low, as shown in Table XI-*A*.

TABLE XI—*A*.

Time of experiment, 104 hours. Distance between electrodes, 1.75 inch (4.5 cm.). Current density, 40 amp. per sq. foot (4.4 amp. per sq. dm.). Electrolyte contained 15 percent $CuSO_4.5H_2O$, 10 percent H_2SO_4. Temperature of electrolyte, 50° C. Anode contained 0.96 percent As and 0.96 percent Sb.

Number of Cells	Percent of As in Electrolyte	Percent of Peptone in Electrolyte	Average E. M. F. in Volt	Impurities in Deposited Copper		Impurities in Elect. after Run		Photo IX-A
				Percent As	Percent Sb.	Percent As	Percent Sb.	
1	3.0	0.01	0.38	0.0478	0.0035	3.31	0.0026	1
2	6.0	0.01	0.40	0.0521	0.0029	6.10	0.0026	2
3	3.0	0.02	0.39	0.0552	0.0057	3.12	0.0016	3
4	6.0	0.02	0.39	0.0437	0.0025	6.21	0.0032	4

As to the amount of impurities in the deposits, they all ran high, both in arsenic and antimony, which impaired the physical properties of the deposited copper.

According to the results of this experiment, peptone alone is not at all a satisfactory "addition-agent" and is, therefore, not suitable to use in electrolytic refining. With a cupric sulphate electrolyte it not only causes the injurious impurities to deposit with the copper, but the copper is also deposited as individual crystals and dendrites, which form a very brittle mass.

Electrolytes Which Contained Organic and Inorganic "Addition-Agents."

In this series of experiments the composition of electrolytes was the same as the composition of those which contained an organic "addition-agent," but with the presence also of an inorganic "addition-agent."

Experiment XI-B.

This experiment was performed in the same way and with the same amounts of peptone added to the electrolytes at the same intervals of time as Experiment XI-*A*, but with an

amount of sodium chloride added, to give the presence of 0.01 percent Cl. The results are given in Table XI-*B*, and the character of the deposit is shown in Photograph XI-*B*.

TABLE XI—*B*.

Time of experiment, 104 hours. Distance between electrodes, 1.75 inch (4.5 cm.). Current density, 40 amp. per sq. foot (4.4 amp. per sq. dm.). Electrolyte contained 15 percent $CuSO_4.5H_2O$, 10 percent H_2SO_4, and 0.01 percent Cl as NaCl. Temperature of electrolyte, 50° C. Anode contained 0.96 percent As and 0.96 percent Sb.

Number of Cells	Percent of As in Electrolyte	Percent of Peptone in Electrolyte	Average E. M. F. in Volt	Impurities in Deposited Copper		Impurities in Elect. after Run		Photo XI-B
				Percent As	Percent Sb.	Percent As	Percent Sb.	
1	3.0	0.01	0.48	0.0011	0.0002	3.24	0.0026	1
2	6.0	0.01	0.49	0.0020	0.0001	6.10	0.0022	2
3	3.0	0.02	0.47	0.0008	0.0002	3.36	0.0013	3
4	6.0	0.02	0.49	0.0017	0.0001	6.18	0.0008	4

It is rather surprising that the deposits obtained from electrolytes containing the combined "addition-agents" of peptone and sodium chloride were very satisfactory, while those obtained from electrolytes containing peptone alone were brittle, very rough and impure. A comparison of the deposits shown in Photograph XI-A and XI-B will show the difference in their character. With the sodium chloride and peptone, the deposits were very good, as regards their physical and chemical properties: they were hard, bright, smooth and finely crystalline. As to their ductility, they were found to be less ductile than those obtained from electrolytes which contained the same amount of sodium chloride alone, as "addition-agent," as the test-strips were broken in two pieces, except No. 3, which cracked almost in two when they were bent double. The chemical analyses show that their purity was exceptionally high in all cases, and that none of them contained more than 0.002 percent arsenic and 0.0002 percent antimony.

Thus it is evident that the presence of a small amount of sodium chloride counteracted the bad or injurious effect of peptone and that the combined action of these two "addition-agents" was beneficial, since it rendered the deposits smooth, solid and pure. Moreover, peptone with the presence of sodium chloride

seemed to increase the hardness of the deposits, and, as evidence, there may be cited the fact that the copper deposited at the beginning of the electrolysis was so hard that it caused the starting-sheets to bend inward, top and bottom.

Experiment XII.

This experiment was conducted in two series, A and B. The combined "addition-agents" for the former series were gelatine and sodium chloride, and those for the latter series were tannin and sodium chloride. The electrolytes for both series were to contain, at the start, the same proportion of organic "addition-agents" as in Experiment X, that is, one part by weight, in 10,000 parts (0.01 percent), and one part in 5,000 parts (0.02 percent) of electrolyte, and 0.01 percent Cl, as sodium chloride. The composition of the electrolytes and the results obtained are recorded in Table XII, and the character of the deposits is shown in Photograph XII.

The deposits from series A, that is, from electrolytes which contained the combined "addition-agents" of gelatine and sodium chloride, were in every case very satisfactory and similar to those obtained from electrolytes containing gelatine alone as "addition-agent." They were very bright, exceedingly solid, perfectly smooth and finely crystalline, and the results of bending tests showed that they possessed very high ductility, as the test-strips, when beaten double, did not even show signs of cracking.

The analyses show that the amount of impurities in the deposited copper was, in each case, exceedingly small, running from 0.0007 percent to 0.0015 percent arsenic, and from 0.0002 percent to 0.0003 percent antimony, which is far below the required limit for the best commercial copper.

Here the fact (as has been pointed out in Experiment X) may be recalled that small dendritic "trees" were formed at the beginning of the electrolysis, when gelatine was alone present in the electrolytes. But it was found in this experiment, with sodium chloride present with the gelatine, that no such "trees" were formed, and on the other hand, the copper first deposited was perfectly smooth. This brings out the fact that the presence of sodium chloride hindered the formation of dendritic "trees," which

would have formed if the sodium chloride were absent and gelatine alone was present in the electrolytes. From this it will be seen that an addition of a small amount of sodium chloride to the electrolytes is quite necessary and important, if gelatine is used to improve the copper deposits.

TABLE XII.

Time of experiment, 104 hours. Distance between electrodes, 1.75 inch (4.5 cm.). Current density, 40 amp. per sq. foot (4.4 amp. per sq. dm.). Electrolyte contained 15 percent $CuSO_4.5H_2O$, 10 percent H_2SO_4, and 0.01 percent Cl as NaCl. Temperature of Electrolyte, 50° C. Anode contained 1.17 percent As and 1.32 percent Sb.

Number of Cells	Percent of As in Electrolyte	Addition-Agent		Average E. M. F. in Volt	Impurities in Deposited Copper		Impurities in Elect. after Run		Photo XII
		Percent Gelatine	Percent Tannin		Percent As	Percent Sb.	Percent As	Percent Sb.	
Series A									A
1	3.0	0.01	. .	0.48	0.0008	0.0002	3.38	0.0054	1
2	6.0	0.01	. .	0.47	0.0015	0.0003	6.51	0.0043	2
3	3.0	0.02	. .	0.46	0.0007	0.0003	3.32	0.0054	3
4	6.0	0.02	. .	0.50	0.0009	0.0003	6.28	0.0023	4
Series B									B
1	3.0	. .	0.01	0.43	0.0014	0.0002	3.91	0.0016	1
2	6.0	. .	0.01	0.44	0.0024	0.0002	6.47	0.0013	2
3	3.0	. .	0.02	0.44	0.0011	0.0003	3.38	0.0019	3
4	6.0	. .	0.02	0.43	0.0026	0.0002	6.49	0.0033	4

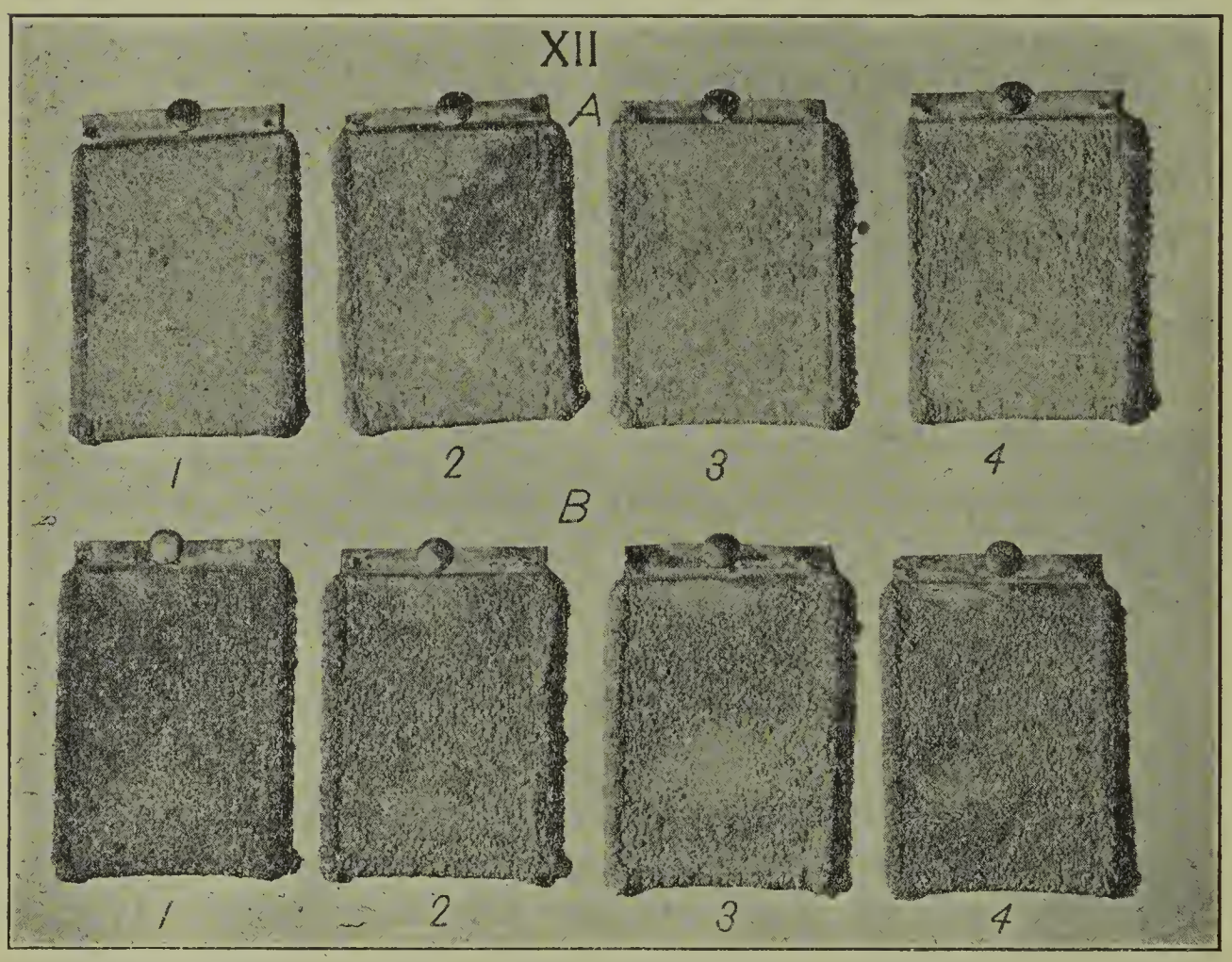
The potential difference was observed to be high with the presence of gelatine in the electrolytes, but as electrolysis continued it gradually dropped, as in the previous experiment. After 80 hours electrolysis a second addition of the organic "addition-agents" was made in both series, A and B, and the amounts added in both cases were the same as originally present.

The deposits from series B, that is, from electrolytes which contained tannin and sodium chloride, were also satisfactory, but to a slightly less degree than those obtained in the above case, as they consisted of coarse but coherent grains with sharp edges. Their physical properties, however, were similar to those of series B, Experiment X, that is, those from electrolytes which contained, as "addition-agent," only tannin. With sodium chloride present their ductility was more or less diminished, for

test-strips from all deposits except No. 3, series B, were broken, when they were hammered double.

Though the presence of sodium chloride with tannin in the electrolytes decreased the ductility of the deposits, it did not seem to affect their purity to any appreciable extent, as the analyses show that they were all very low in arsenic and antimony. The potential difference was lower than that of series A, and remained practically constant throughout the run.

From what has been said in regard to series B, it will be seen



that an addition of sodium chloride does not seem necessary when tannin is used, for tannin alone appears to improve the physical properties of the deposits more effectively than when sodium chloride is also an associate.

Experiment XIII-A.

In this experiment the combined "addition-agents" used were peptone and hydrochloric acid, and gelatine and hydrochloric acid. The object of using hydrochloric acid was to ascertain the

effect of Cl ion in the presence of either of the organic substances mentioned. The electrolysis was conducted in four cells, two for each of the combined "addition-agents." The electrolytes were made in each case to contain, at the start, one part, by weight, of the organic "addition-agent" in 5,000 parts of the electrolyte (0.02 percent) and 0.01 percent Cl, as hydrochloric acid, the preparation of which was made in the same manner as in Experiment VI. The composition of the electrolytes and the results obtained are expressed in Table XIII-A. and the character of the deposits is shown in Photograph XIII-A. Deposits No. 1 and No. 2 were obtained from electrolytes containing peptone and hydrochloric, and deposits Nos. 3 and 4 from electrolytes containing gelatine and hydrochloric acid.

TABLE XIII—*A*.

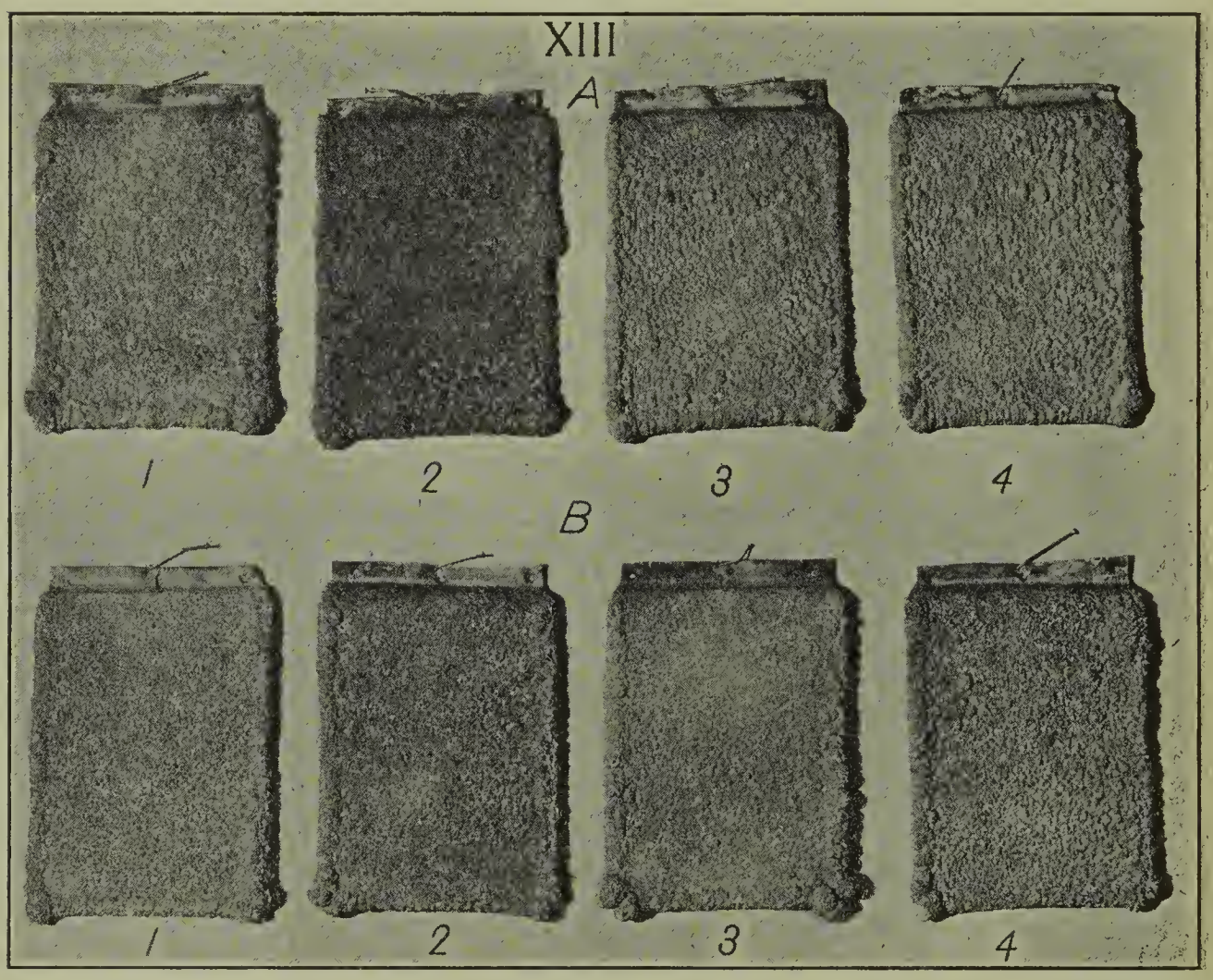
Time of experiment. 104 hours. Distance between electrodes, 1.75 inch (4.5 cm.). Current density, 40 amp. per sq. foot (4.4 amp. per sq. dm.). Electrolyte contained 15 percent $CuSO_4$, 10 percent H_2SO_4, and 0.01 percent Cl as HCl. Temperature of electrolyte, 50° C. Anode contained 1.30 percent As and 1.27 percent Sb.

Number of Cells	Percent of As in Electrolyte	Percent of Gelatine	Percent of Peptone	Average E. M. F. in Volt	Impurities in Deposited Copper		Photo XIII-A
					Percent As	Percent Sb.	
1	3.0	. .	0.02	0.52	0.0028	0.0003	1
2	6.0		0.02	0.49	0.0030	0.0004	2
3	3.0	0.02	.	0.48	0.0016	0.0003	3
4	6.0	0.02	. .	0.49	0.0022	0.0002	4

During the electrolysis it was observed that the potential difference in each case was high and about the same as that of Experiment XI-B and Experiment XII, when gelatine and sodium chloride were used. And, in the same way, the potential difference gradually dropped as electrolysis proceeded. This drop of voltage, it is believed, ascertained the consumption of the organic substance. Three further additions of the organic "addition-agents" were made in each case during the run, at such intervals as deemed necessary, in order to make up their consumption.

With peptone and hydrochloric acid in the electrolytes it was observed that the copper first deposited was perfectly smooth and

so hard, as in the case where sodium chloride and peptone were present, that the starting-sheets were bent slightly inward, top and bottom. But in the course of about fifteen hours, small rounded nodules began to form, thinly scattered on the surface of the cathodes. As the electrolysis continued, the bright color of the deposits gradually, but slightly, changed, and began to appear more and more dull. Toward the end of the run the surface of both deposits became quite dull. In the case in which gelatine and hydrochloric acid were added to the electrolytes no



such action was observed and, on the contrary, the deposits formed brighter and more smooth throughout the electrolysis.

Deposits No. 1 and No. 2, as shown in Photograph XIII-*A*, that is, deposits from electrolytes which contained as "addition-agents" peptone and hydrochloric acid, were exceedingly hard, coherent, but rough and of dull color. They were composed of fine grains, but with large rounded nodules scattered over their surface. The bending test showed that the ductility of these two deposits was inferior to that of those obtained from electrolytes

containing peptone and sodium chloride, as the test-strips were broken when they were slightly bent. The impurities in the deposits were low.

Deposits No. 3 and No. 4, that is, deposits from electrolytes containing gelatine and hydrochloric acid as "addition-agents," were however satisfactory; they were bright and fairly smooth, solid and coherent. The bending test showed their ductility to be very low, about the same as that of cathodes No. 1 and No. 2 of this experiment. The purity of the deposits was high, as shown in Table XIII-*A*.

The results of this experiment show that the combined "addition-agents" of peptone and hydrochloric acid had a somewhat similar behavior to combined peptone and sodium chloride, but less satisfactory and less effective.

The combined "addition-agents" of gelatine and hydrochloric acid were very effective in improving the physical properties of the deposits and preventing the deposition of the impurities, but were not so effective as the combined gelatine and sodium chloride, which proved the most satisfactory and suitable of all the combined "addition-agents" which were employed in these experiments.

Experiment XIII-B.

In order to see if the addition to the electrolytes of sodium salts, which consist of an oxidizing acid radical, would produce any bad effect upon the copper deposit, this experiment was performed with electrolytes containing 1.5 percent arsenic, as arsenic acid, and the same proportions of cupric sulphate and sulphuric acid as in the previous experiments. The sodium salts tried were sodium chloride, sodium nitrate, sodium chlorate and sodium borate. The amount of salt added to the electrolyte was such that it contained, in each case, 0.01 percent sodium. The results are given in Table XIII-*B* and the character of the deposits is shown in Photograph XIII-*B*.

With sodium chloride in the electrolyte, the deposit was lustrous, perfectly smooth, solid and coherent, and was composed of crystals with sharp edges, while the deposits formed from electrolytes contained sodium nitrate, sodium chlorate, and sodium borate were also satisfactory and similar to one another in char-

acter. They were bright, solid, smooth, coherent and finely crystalline, though at the lower corners of deposit No. 3 were formed a few large nodular crystals. As to the ductility of these four deposits, those obtained from electrolytes containing sodium nitrate, sodium chlorate, and sodium borate were ductile, as the test-strips, when beaten double, did not break. The test-strip of the deposit from the electrolyte containing sodium chloride broke when it was hammered double, and its fracture appeared fibrous. The purity of all the deposits was exceptionally high, as may be observed in Table XIII-*B*.

TABLE XIII—*B*.

Time of experiment, 104 hours. Distance between electrodes, 1.75 inch (4.5 cm.). Current density, 40 amp. per sq. foot (4.4 amp. per sq. dm.). Electrolyte contained 15 percent $CuSO_4.5H_2O$, 10 percent H_2SO_4, and 1.5 percent As as H_3AsO_4. Temperature of electrolyte 50° C. Anode contained 1.30 percent As and 1.27 percent Sb.

Number of Cells	Addition-Agents	Average E. M. F. in Volt	Impurities in Deposited Copper		Photo XIII-B
			Percent As	Percent Sb.	
1	0.01 Percent Na as NaCl	0.40	0.0009	0.0002	1
2	0.01 Percent Na as $NaNO_3$	0.42	0.0018	0.0002	2
3	0.00 Percent Na as $NaClO_3$	0.41	0.0019	0.0004	3
4	0.01 Percent Na as $Na_2B_4O_7$	0.45	0.0010	0.0002	4

During the electrolysis the potential difference in the case of sodium borate was noted to be little higher than that of the rest.

According to the results obtained from this experiment, the sodium salts consisting of an oxidizing acid radical, such as those mentioned above, do not appear to exert any injurious influence upon the deposited copper, when present in small amounts. On the other hand they show themselves to be beneficial "addition-agents," instead of detrimental, as it was found that they improved to a considerable extent the purity as well as the physical properties of the deposited copper.

Experiment XIV.

For this experiment and the following one, the electrolytes contained the same proportions of cupric sulphate and free sulphuric acid as in previous experiments and 3 percent of

arsenic as arsenic acid. The "addition-agents" tried were aluminium chloride, cuprous chloride, cupric chloride and glue. The electrolysis was conducted under the same conditions as those previously performed.

As has been shown in Experiment IX-*A*, the addition of 0.1 percent Al as aluminium chloride caused the separation and precipitation of oxide of arsenic on the cathode at the beginning of the electrolysis. In order to determine whether this action was due to the excessive amount of aluminium chloride, a much smaller addition of this salt to the electrolyte was made in this case; the amount added was 0.005 percent Al as aluminium chloride. It was observed during electrolysis that no such action as occurred in Experiment IX-*A* took place, and the deposit obtained was bright, solid and smooth, but composed of small crystals with sharp edges. It was quite ductile, as the test-strip, when hammered double, only cracked a little at the bend. Photograph XIV, No. 1, shows the character of the deposit.

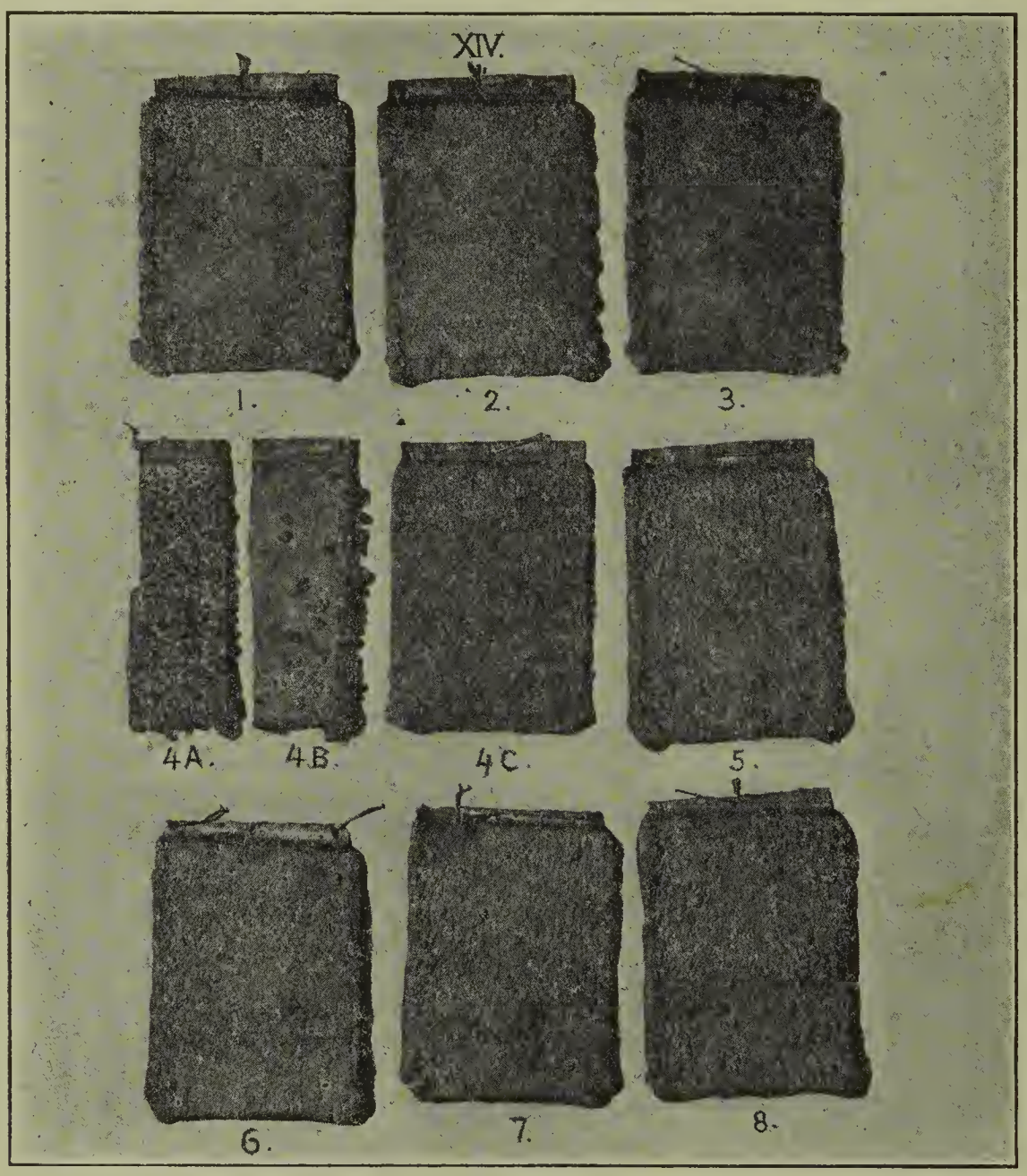
From the results given above it is seen that the presence of an excess of aluminium chloride was found to exert a bad effect upon the deposited copper, especially at the beginning of the electrolysis. In order, then, to produce good and pure copper, it is important that the addition of this salt should be limited to a very small amount.

The deposits which formed in electrolytes containing 0.01 percent Cl as cuprous chloride, and as cupric chloride, were satisfactory and quite similar to each other. Deposits Nos. 2 and 3, Photograph XIV, show the character of the respective deposits. They were bright, fairly solid, finely crystalline and free of "trees." The results of the bending tests showed that they were not ductile, as the test-strips were broken in two when bent. Their color and fracture, however, indicated that they were of high purity.

Glue was another "addition-agent" employed in this experiment in order to find out its effect upon the deposited copper. The solution of glue was prepared in the same manner as in the case of gelatine. The amount added to the electrolyte was 0.01 percent, that is, one part, by weight, of glue to 10,000 parts of electrolyte.

At the beginning of the electrolysis small fern-like "trees"

formed and scattered all over the surface of the cathode. This is shown in Photograph XIV, No. 4-*A*. At the end of fifteen hours the cathode was withdrawn to knock off the "trees" along the edges, and again placed in its original position with the reverse-side front. As electrolysis proceeded similar "trees" again appeared, but greatly decreased in number, as is shown in



Photograph XIV, No. 4-*B*. After forty-three hours the cathode was superseded by a new starting sheet in order to ascertain whether the bad action of glue, after this time, would cease. The copper deposited from now on was observed to be quite smooth, and the formation of "trees" entirely ceased. This deposit is shown in Photograph XIV, No. 4-*C*. It was very bright, exceedingly solid and extremely ductile, as the test-strip

did not crack when hammered double. These properties showed that the copper was of high purity.

From the results mentioned above and those described in Experiment X, series A, it will be seen that glue and gelatine exert a similar influence upon the deposited copper. With gelatine in the electrolyte the formation of small fern-like "trees" ceased after fifteen hours electrolysis, and the copper deposited was from this time on perfectly smooth; while with glue in the electrolyte, the formation of such "trees," though greatly decreased in numbers after the same interval of time, did not entirely cease until the electrolysis had continued for some forty-three hours. Moreover, the ductility of the copper obtained from the electrolyte which contained gelatine seemed to be little greater than that obtained from similar electrolyte containing glue. Since gelatine is refined glue, these differences may be attributed to the impurities which the glue contained.

Experiment XIV-A.

In this experiment the combined "addition-agents" of glue and sodium chloride, glue and hydrochloric acid, glue and cuprous chloride, and glue and cupric chloride, were employed. The electrolyte was in each case made to contain 0.01 percent glue and 0.01 percent Cl as chloride.

The deposits which formed in these electrolytes were in each case very satisfactory and similar to one another in character. They were also similar to those which were obtained from electrolytes containing the combined "addition-agents" of gelatine and sodium chloride, and gelatine and hydrochloric acid. They are very bright, exceedingly solid, quite smooth and finely crystalline, though a few small rounded nodules formed and scattered on the surfaces of the deposits. As the results of bending tests, they were found to be very ductile, as the test-strips when hammered double did not show any sign of cracking. The character of these deposits is shown in Photograph XIV, as follows:

No. 5 deposit, from electrolyte containing glue and hydrochloric acid.
No. 6 " " " " " " sodium chloride.
No. 7 " " " " " " cuprous chloride.
No. 8 " " " " " " cupric chloride.

Conclusions.

The experiments, as performed, may be grouped under four main headings: (1) Impure electrolytes which contained no "addition-agents." (2) Impure electrolytes which contained inorganic "addition-agents." (3) Impure electrolytes which contained organic "addition-agents." (4) Impure electrolytes which contained combined organic and inorganic "addition-agents."

With electrolytes which contained no "addition-agents" much arsenic and antimony were deposited with the copper, even when the electrolytes contained 1.5 percent arsenic. With electrolytes containing 2 percent and 3 percent arsenic, a still greater amount of these two impurities was deposited with the copper, and large dendritic "trees" also formed on the surface of the deposits. These impurities rendered the copper dull-colored and brittle.

At temperatures between 50° C. and 60° C., and with electrolytes containing over 6 percent arsenic, the arsenic acid appeared to act as an "addition-agent," as it prevented, to some extent, the deposition of the impurities (arsenic and antimony) with the copper, and also retarded the formation of dendritic "trees." At temperatures of 40° C. and below, however, this action of arsenic acid as "addition-agent" in the electrolyte did not appear to take place in the electrolyte containing under 6 percent arsenic, whereas in the electrolyte containing 8 percent the deposited copper was purer, brighter, more solid and coherent, and less brittle. This shows that the good effect of arsenic acid depends not only upon the temperature of the electrolyte, but also upon the amount which is present in the electrolyte. This phenomenon cannot be explained from the data obtained in these experiments and without further investigations.

Hydrochloric acid, cuprous chloride, cupric chloride, aluminium chloride, sodium chloride, sodium sulphate, sodium nitrate, sodium chlorate, sodium borate and aluminium sulphate, all, when present in small amounts, cause a distinct improvement of the deposited copper, both chemically and physically; the deposits become more or less smooth, dense, purer, and free from "trees." Of these inorganic "addition-agents" the best and most effective is sodium chloride; whereas hydrochloric acid, cupric chloride and cuprous chloride are so to a slightly less degree. The effect

of sodium sulphate is still less; it produces little or no effect if present in too small amounts, especially at temperature of 40° C. and when the electrolyte contains 3 percent arsenic. Aluminium chloride, unless present in amounts of not more than 0.005 per-cent, does not seem to be a suitable "addition-agent" because, as has already been pointed out, the presence of a large amount (0.1 percent Al) causes the separation and precipitation of oxide of arsenic on the cathode at the beginning of the electrolysis. Aluminium sulphate was found, on the other hand, to be a satisfactory "addition-agent," even when present in large amounts, causing the copper *to deposit with less impurities and quite ductile.*

From the results of the trial of the inorganic "addition-agents" it appears to be true that the salts of those metals which stand far higher in the e. m. f. series than copper are generally satisfactory "addition-agents." They possess the property of preventing, to a considerable extent, the deposition of arsenic and antimony and the formation of "trees." The latter action of inorganic "addition-agents" is difficult to explain, but according to Edward F. Kern[14] it is due to the reducing effect of the metallic ions which, by the aid of the electric current, form a menstruum around the cathode; in other words, this menstruum of metallic ions has the influence, or property, of more or less changing the copper crystals to an amorphous form. The higher the metal of the "addition-agent" stands in the e. m. f. series, the purer, smoother and less brittle the deposit forms.

The Chemical Behavior of the Na Ion in the Electrolyte.

As the physical action of the Na ion has just been mentioned, it now becomes necessary to explain its chemical behavior, that is, why the presence of Na ion prevents the deposition of arsenic with the copper. This phenomenon may, however, be explained as follows: It is a known fact that during electrolysis the arsenic in the anode passes into solution as arsenous sulphate, $As_2(SO_4)_3$, which is hydrolysed, forming, in an acid solution, arsenous hydroxide, $As(OH)_3$, behaving as a base. This new substance is slightly dissociated according to the following scheme:

$$As(OH)_3 = \dddot{A}s + 3(OH)'$$

[14] Tran. Amer. Electrochemical Society, **15**, 473 (1909).

If, then, this is true, the law of mass action may be applied, that is,

$$\frac{C_{(As)} \times C^3_{(OH)}}{C_{[As(OH)_3]}} = \text{constant (K)}.$$

where $C_{(As)}$ is the concentration of $\dddot{A}s$ ion, $C_{(OH)}$ the concentration of OH'. ion, and $C_{[As(OH)_3]}$ the concentration of the undissociated $As(OH)_3$.

When the undissociated $As(OH)_3$ comes in contact with the $\dot{N}a$. ion which surrounds the cathode or thereabout, the $\dot{N}a$. ion converts the arsenious hydroxide into a new compound (Na_3AsO_3), the chemical reaction of which may be shown as follows:

$$As(OH)_3 + 3\dot{N}a = Na_3AsO_3 + 3\dot{H}$$

that is, the $\dot{N}a$. ion in the electrolyte replaces the hydrogen, setting it free as $\dot{H}$. ion, and at the same time changing the arsenic cation into a complex anion $(AsO_3)'''$, as is seen in the above equation. From this it appears that the presence of $\dot{N}a$. ion diminishes the amount of the undissociated $As(OH)_3$. If the concentration of the undissociated $As(OH)_3$ is decreased, then the product of $C^{(As)}$ and $C_{(OH)}$, according to the laws of mass action, must be simultaneously decreased, in other words, some of the $\dot{A}s$. ion and OH'. ion must unite to form undissociated $As(OH)_3$ in order to give the constant. This seems to indicate that the $\dot{N}a$. ion indirectly decreases the amount of the positive As ion, thus preventing it from wandering to the cathode and depositing there with the copper.

The Behavior of Cl'. Ion in the Electrolyte.

It may be recalled here that without the presence of Cl'. ion in the electrolyte the deposits became rough and contained a large proportion of arsenic, which impairs the electrical conductivity, the malleability and the ductility of copper. The deposition of arsenic on the cathode is without doubt due to the arsenic which exists in the electrolyte as cation, and its existence may be attributed to the fact that, as above explained, arsenious hydroxide, $As(OH)_3$, is formed, which is slightly ionized into arsenic cation and hydroxyl anion. The former, being a positive

ion, goes to the cathode under the action of the electric current and there it deposits with the copper, thus rendering the deposited copper impure.

Now, if an addition of a small amount of Cl′. ion to the electrolyte is made, a different phenomenon is observed; the deposited copper is thereby rendered purer and more solid. This, therefore, demonstrates that the presence of even a very small amount of Cl′. ion in the electrolyte prevents, to a considerable extent, the deposition of arsenic on the cathode. This phenomenon is probably brought about by the oxidizing action of the Cl′. ion, which surrounds the anode, converting the arsenic from the cationic into the anionic state. This may take place as follows: The arsenic contained in the anode, as previously mentioned, dissolves during electrolysis as arsenous sulphate, $As_2(SO_4)_3$. It is then hydrolysed, forming arsenous hydroxide, $As(OH)_3$. But, with the presence of Cl′. ion, the hydrolysis of this salt is prevented, as the Cl′. ion surrounding the anode oxidizes the arsenous sulphate to arsenic sulphate at the moment when the arsenous sulphate is formed. The chemical reaction may be represented by

$$As_2(SO_4)_3 + 2Cl'_2 + 2H_2SO_4 = As_2(SO_4)_5 + 4HCl.$$

It will be seen from this equation that the chlorine, after oxidation, is not lost, as hydrochloric acid is generated, which is again dissociated into H. and Cl′. ions. It appears, then, that the Cl′. ion only serves the purpose of effecting the oxidation of arsenous sulphate, and therefore, theoretically, a small quantity of Cl′. ion present in the electrolyte should be capable of transforming an infinite amount of arsenous sulphate into arsenic sulphate, as HCl is reformed.

The arsenic sulphate, thus formed, reacts with the water forming arsenic acid; thereby, the arsenic being converted from the cationic to the anionic condition. The reaction may be expressed according to the following equation:

$$As_2(SO_4)_5 + 8H_2O = 2H_3AsO_4 + 5H_2SO_4.$$

From the two chemical equations mentioned above it appears that the presence of Cl′. ion in the electrolyte is of much import-

ance, as it converts the arsenic cation into the anion, and thus prevents the deposition of arsenic with the copper.

It has been pointed out that sodium chloride is a better and more effective "addition-agent" than any of the other salts, or acids, which contain either sodium or chlorine as one of their constituents. If the above explanations of the behavior of the $\dot{Na}$. and Cl′. ions are correct, the more effective action of sodium chloride must be due to the beneficial influence of both the $\dot{Na}$. and the Cl′. ions.

An excess of chlorine ions in the electrolyte causes the deposits to become brittle and less pure. It is therefore of first importance that the amount of chloride added should be limited to a very small quantity, that is, to about 0.01 percent of chlorine as chloride, preferably sodium chloride. A smaller amount is also beneficial.

Temperature plays an important part in the electrolysis of copper solutions. With higher temperature the ductility of the copper seems to be increased and the potential between the electrodes decreases. In the case of sodium sulphate as "addition-agent," it is more effective the higher the temperature of the electrolyte.

The Effect of Organic "Addition-agents" in the Electrolyte.

In the case of organic "addition-agents," gelatine, glue and tannin produce a remarkably beneficial effect upon the copper deposits, and are satisfactory "addition-agents." The presence of either gelatine or glue at the beginning of the electrolysis has the effect of causing the formation of small fern-like "trees" with rounded ends, but after operating for some time these two organic substances seem to change their property, as it was observed that the formation of "trees" ceased, and that the deposits were smooth and ductile. Gelatine and glue, therefore, exercise a similar effect upon the deposits, and their behavior differs only in that gelatine gives smoother deposits in a much shorter time. Peptone, unlike gelatine and glue, exerts a detrimental influence on the deposits, which with this "addition-agent" are rough, non-coherent and impure; therefore this is unsatisfactory.

To explain the good and bad action of the organic "addition-agents" mentioned above is no easy matter, as their chemical

structure, except tannin, is not yet fully and definitely known. It may, however, be of value to cite their properties. Tannin, $C_{12}H_3(OH)_3(COOH)_2$, possesses characteristic acid properties and contains three hydroxyls per molecule. Gelatine, the chemical structure of which is not yet fully known, is also essentially acid in character, as it possesses, when pure, an acid reaction and dissociates carbonates. Glue is crude gelatine.

"Peptone[15] is of two kinds, which differ from each other in one molecule of H_2O. Their chemical composition may be shown as follows: $C_{22}H_{34}N_6O_9$ and $C_{22}H_{34}N_6O_{10}$; the former is called alpha-peptone and the latter beta-peptone. Peptones are pronounced acids, which redden litmus paper and which form salts with carbonates after having expelled the carbonic acid gas. Adopting the simplest formula, peptones are monobasic acids, but such a simple formula has to be multiplied."

The deposits of copper obtained from electrolytes containing the combined "addition-agents" were all satisfactory, particularly when the combination of gelatine and sodium chloride, glue and sodium chloride, or glue and cuprous or cupric chloride, was used. Of all these combinations, gelatine and sodium chloride appeared to be the best, though the differences among them were very small. The striking effect of the inorganic substance which contains the Cl radical, when present with the organic, is that it counteracted the bad effect of the latter. Of all the inorganic compounds that were used for the combined "addition-agents," sodium chloride is more suitable than the rest, for it is more effective in producing good and smooth, ductile copper. This shows that the better effect is due to the presence of sodium ion in the electrolyte. As evidence a particular case may be cited, that is, a case in which peptone and sodium chloride, and peptone and hydrochloric acid were used as combined "addition-agents." The copper deposits obtained in the former case were found better, both in physical character and in purity.

In considering what has been said above, the questions may now arise: What causes the organic compounds to behave so differently in the electrolytes when an addition of a small amount of Cl′. ion is made? Does the presence of this Cl′. ion cause the organic compounds to undergo a chemical change? These

[15] "Chemistry of Proteids," Mann, p. 188.

questions cannot be answered without special investigations, and are not within the scope of the present research.

With the results that have been obtained, and with the effects noted of various "addition-agents" tried, this may be said: The combined "addition-agents" of gelatine and sodium chloride, and of glue and sodium chloride, when present in small amounts, are the most suitable and most satisfactory "addition-agents" for copper sulphate electrolytes, containing especially a high proportion of arsenic, as with these the deposited copper possesses the greatest ductility and the highest purity. But the combined "addition-agent" of gelatine and sodium chloride seems to be a little better than sodium chloride and glue, for it gave smoother deposits. In the case of glue and inorganic compounds, the use of cuprous chloride, cupric chloride or hydrochloric acid is just as effective as sodium chloride; but for economic reasons, sodium chloride may be preferred.

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School of Mines, Columbia University,
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DISCUSSION.

Mr. W. L. Spalding (*Communicated*): Experiments are being made upon the effect of adding sodium chloride to the electrolyte under the usual working conditions of a copper refinery. While not carried to completion, they have brought out several points which may be of interest in connection with this paper. Four of the ordinary tanks were provided with an independent heating and circulating system, and were filled with electrolyte containing about 2 percent of arsenic, and kept at a temperature varying from 55° to 50° C. The amount of sodium chloride necessary to increase the chlorine to 0.010 percent was calculated to be about 3 lb. (1.5 kg.). This amount did not give the desired content, and more additions were made at intervals, until at the end of two months 18 lb. (8 kg.) had been added, and the percentage of chlorine was still about 0.005. It was found that

the chlorine slimes as cuprous chloride. Presumably this was brought about either by the reduction of the cupric to cuprous chloride by the metallic copper, or more probably by the electro-chemical corrosion of the anode by the chlorine ions, as in a small cell with considerable salt in the electrolyte the anode was covered with a thick, slimy layer of whitish Cu_2Cl_2. The cathodes from these tanks were dark and crumbly and much higher in arsenic (some as high as 0.03 percent) than those in the neighboring tanks, which were filled with the same electrolyte, but to which salt had not been added.

Dr. E. F. Kern (*Communicated*): The results of Mr. W. L. Spalding's experiments are very interesting; but there is not sufficient data given to permit an answer being given as to the cause of the formation of cuprous chloride on the anodes. It has been found that when copper is refined in a sulphate electrolyte to which a chloride has been added, the cuprous chloride which is formed on the anode increases as the free acid in the electrolyte is increased, and also as the current density is decreased. Temperature and circulation also influence the formation of the insoluble cuprous chloride; the higher the temperature and the greater circulation, the less of the insoluble salt on the anode. Probably in the case of Mr. Spalding's experiments the acid content of the electrolyte was above 12 percent, and the current density was below 15 amperes per square foot. A change of these conditions may give more satisfactory results.

A paper read in abstract by F. A. Lidbury, at the Twentieth General Meeting of the American Electrochemical Society, in Toronto, Canada, September 21, 1911, President W. R. Whitney in the Chair.

THE CONDUCTIVITY OF MIXTURES OF COPPER SULPHATE AND SULPHURIC ACID

By H. K. Richardson and F. D. Taylor.

To obtain figures for calculating the voltage drop due to electrolytic resistance in a copper refining tank, assumptions must be made regarding the conductivity of mixtures of sulphuric acid and copper sulphate. The only experimental results published are very fragmentary, those of Addicks[1] being untrustworthy, his curves not agreeing within 10 percent amongst themselves, and his value for the resistance of 8 percent sulphuric acid being about 30 percent off. Those of Thompson and Hamilton[2] are for only two limiting values. These results agree with ours to within 2 percent.

The results given in this paper are only part of those which it was desired to obtain regarding the effect of various impurities upon the resistance of the refinery electrolyte. Owing to circumstances, no further work could be done, and the results so far obtained seemed interesting enough to publish.

METHOD.

The conductivity of the various mixtures was determined by the standard Kohlrausch method, using a cell of construction shown in Fig. 1.

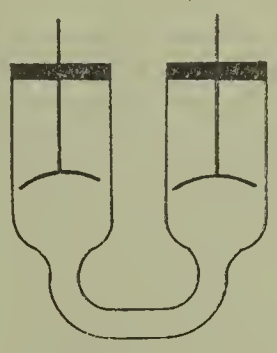


Fig. 1.

[1] Elect. Met. Industry, **4**; 17, Fig. 2 (1906).
[2] Trans. American Electrochemical Soc., **17**, 292 (1910).

All solutions were made up by measuring out from stock solutions of copper sulphate and sulphuric acid the volume wanted, and diluting same up to one liter (1,000 c.c.). In case of the mixtures carrying 20 grams of $CuSO_4,5H_2O$ per 100 c.c. it was necessary to add a weighed amount of the material.

The chemicals used were from the J. T. Baker Co., and had the following analysis on the labels:

Special $CuSO_4$, $5H_2O$.
Iron = 0.0003%.
Chloride = None.

H_2SO_4.
Sp. Gr. 1.84.
H_2SO_4 = 95.6 — 96.4%
N.V.M. = 0.0008%.
Iron = 0.0004%.
HCl, HNO_3, As = None.

RESULTS.

The results obtained are tabulated below.

TABLE I.

Conductivity Expressed in Reciprocal Ohms per Centimeter Cube.

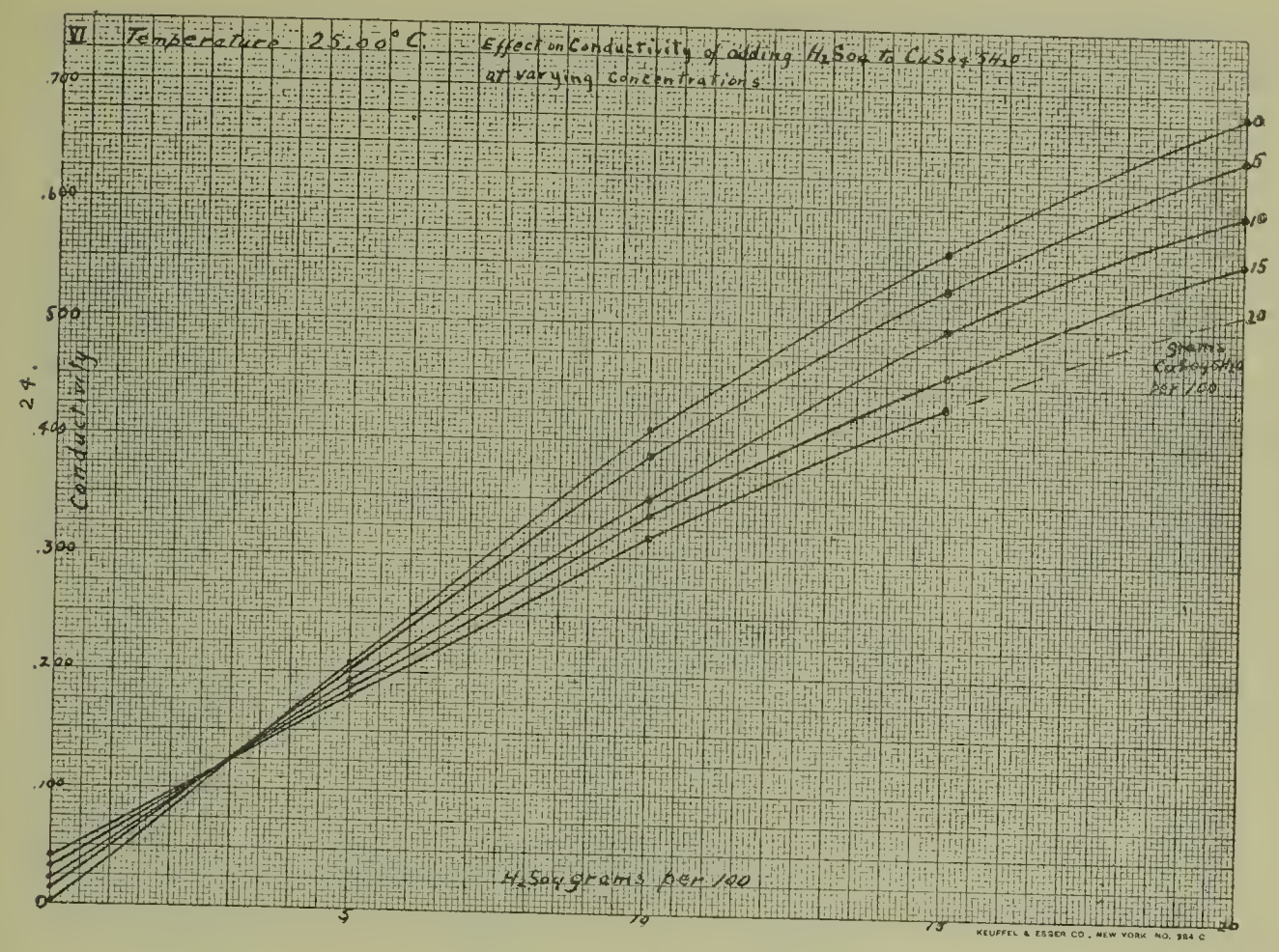
H_2SO_4 Grams per 100 c.c.		25°C					45°C				
		0	5	10	15	20	0	5	10	15	20
$CuSO_4 \cdot 5H_2O$ Grams per 100 cc.	0 . .	. . . .	0.208	0.410	0.565	0.683	. . . .	0.246	0.492	0.683	0.839
	5 . .	0.0153	0.204	0.388	0.531	0.646	0.0205	0.242	0 461	0.643	0 791
	10 . .	0.0221	0.195	0.350	0.500	0.600	0.0294	0.222	0.422	0.606	0.738
	15 . .	0.0348	0.189	0.338	0.458	0.558	0.0468	0.217	0.381	0.545	0.690
	20 . .	0.0423	0.182	0.319	0.433	. .	0.0574	0.212	0.378	0.521	0.643

These results are plotted in Figs. 2-5.

To obtain the values of above in terms of resistance per inch cube, take the reciprocal and multiply by 0.394, as,

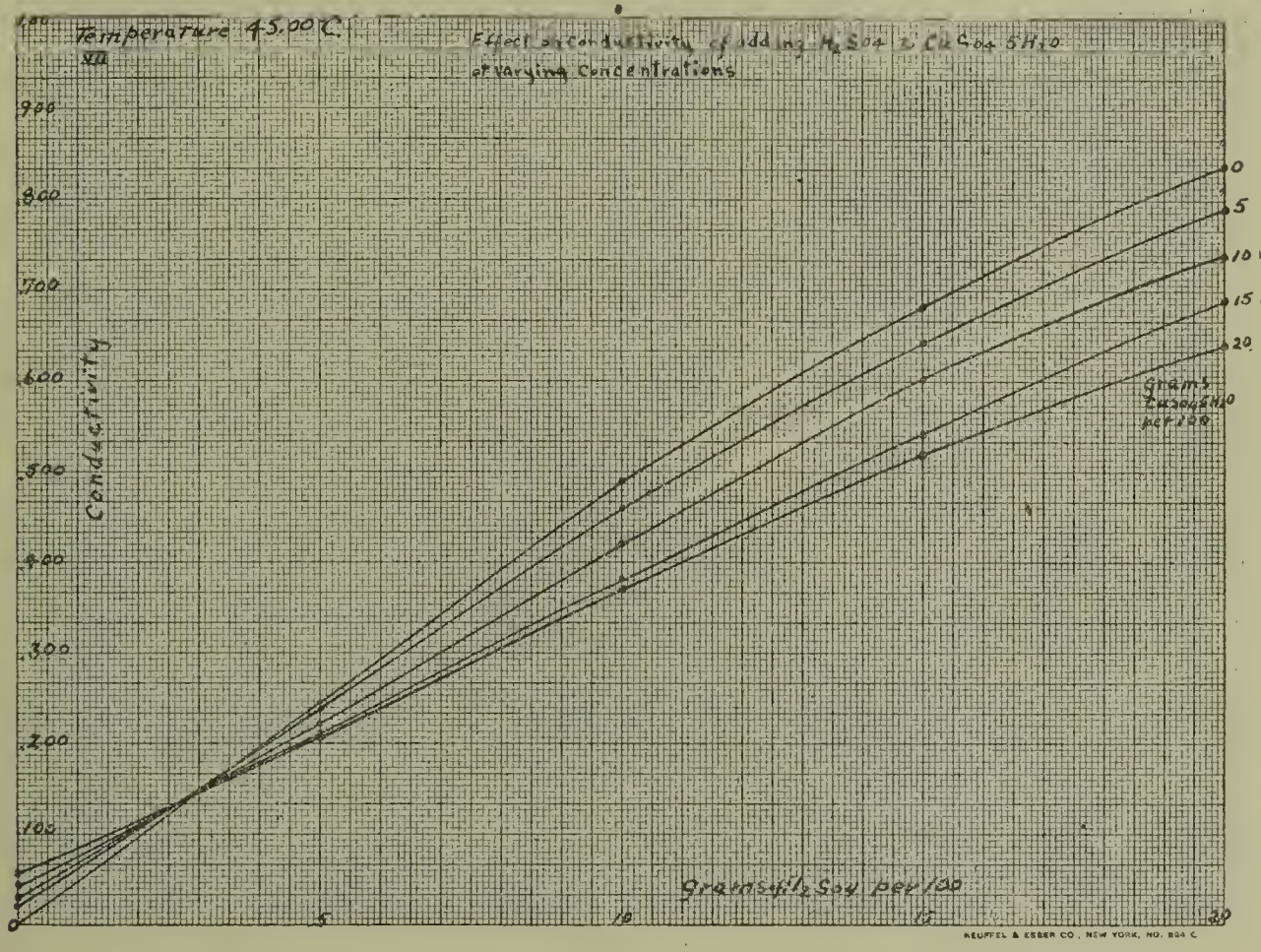
Ohms per inch cube = 0.394 × ohms per centimeter cube.

In practical work, most all strengths of solutions are given in terms of percentages. To convert the values in Table I to these units, use Table II.


VI Temperature 25.00° C. Effect on Conductivity of adding H_2SO_4 to $CuSO_4 5H_2O$ at varying Concentrations.
Conductivity
H_2SO_4 grams per 100
Grams $CuSO_4 5H_2O$ per 100
0
5
10
15
20
KEUFFEL & ESSER CO., NEW YORK NO. 384 C

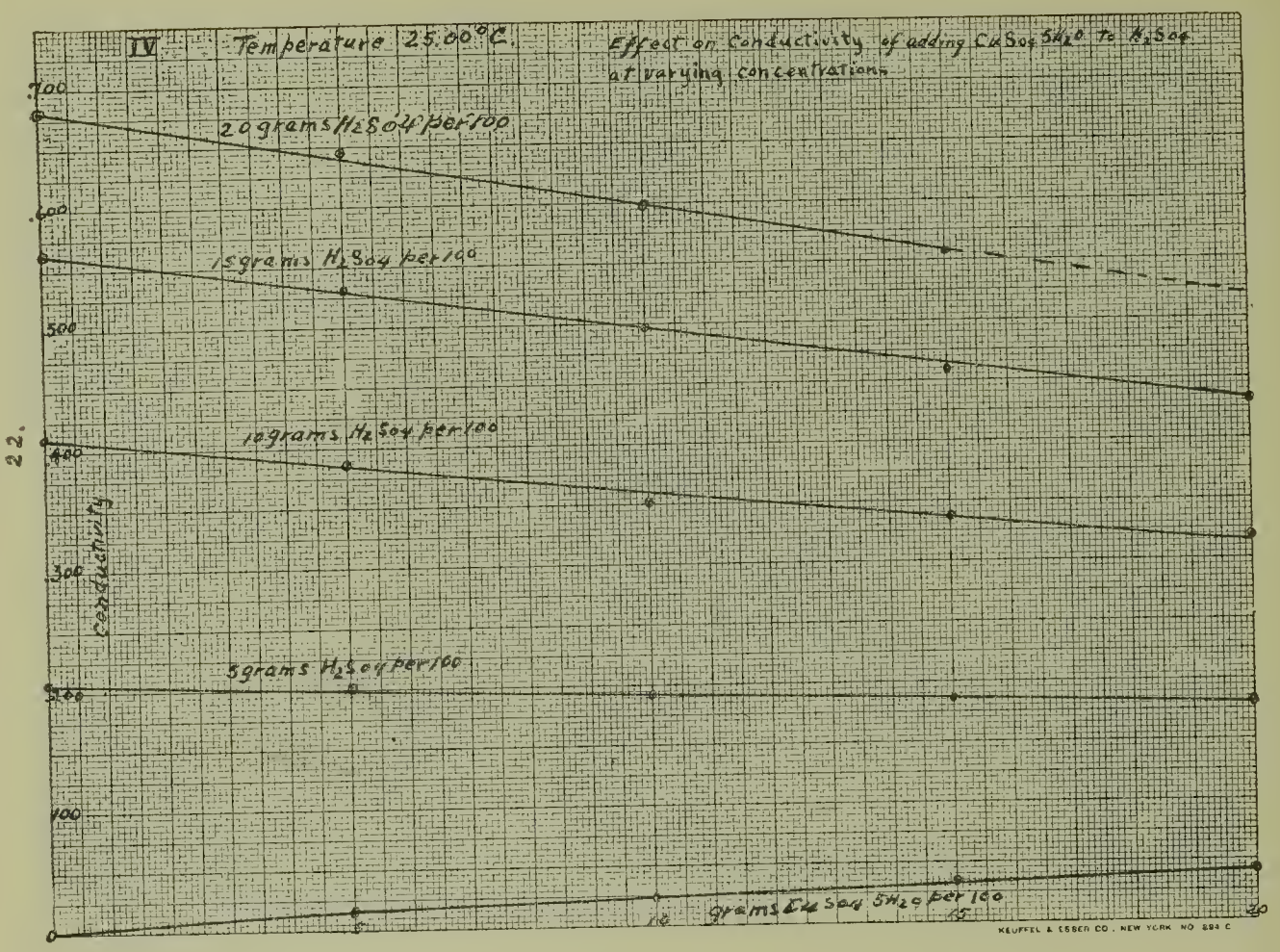
24.

FIG 2.



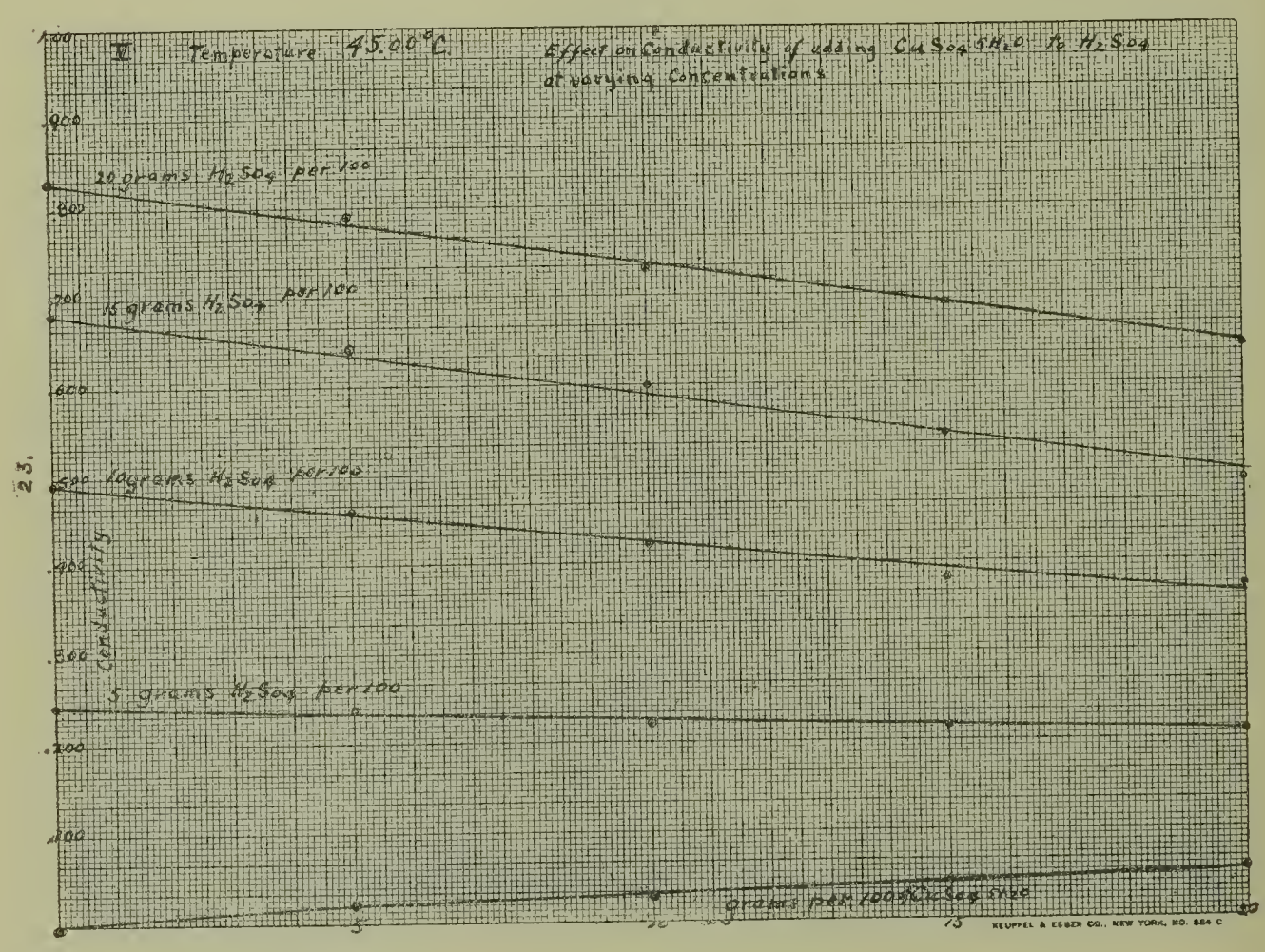
25.

FIG. 3.



22.

FIG. 4.



23.

FIG. 5.

TABLE II.

Percentage			Grams per 100 c.c.			Gram Equiv. per 1000 cc.	Percent H_2SO_4	Grams per 100 c.c.	Grams Equiv. per 1000 c.c.
Copper	$CuSO_4$	$CuSO_4 \cdot 5H_2O$	Copper	$CuSO_4$	$CuSO_4 \cdot 5H_2O$				
1.00	2.5	3.91	1.04	2.56	4.0	0.321	5	5.15	1.05
2.00	5.0	7.82	2.09	5.25	8.2	0.658	10	10.7	2.18
3.99	10.0	15.64	4.41	11.05	17.4	1.387	15	16.5	3.38
5.99	15.0	23.46	6.97	17.50	27.4	2.194	20	22.8	4.66
6.98	17.5	27.37	8.37	22.00	32.9	2.631			

TEMPERATURE COEFFICIENT.

From an analysis of Table I we get the following approximate temperature coefficients for the range 25°-45° C.

TABLE III.

Coefficient Expressed in Percentage per Degree Centigrade.

Grams per 100 cc.	$CuSO_4$ Only	H_2SO_4 Only	Mixtures of $\{H_2SO_4, CuSO_4$ Containing equal amounts of each
5	1.70	0.92	0.93
10	1.65	1.00	1.03
15	1.72	1.13	0.95
20	1.78	1.14	. . .
Average for all Concentrations	1.71	1.05	0.97

In practice, the refinery electrolyte is said[3] to have a temperature coefficient of approximately 0.5 percent per degree F., or 0.90 percent per degree Centigrade, which corresponds well with that in the third column above.

CONCLUSIONS.

First.—Addition of copper sulphate to solutions of sulphuric acid—

(*a*) Increases the conductivity of the mixture if the sulphuric acid is less than 3 gms. per 100 c.c.

(*b*) Has no effect on the conductivity of the mixture if the sulphuric acid is 3 gms. per 100 c.c.

[3]Addicks, loco cit., p. 18.

(*c*) Decreases the conductivity of the mixture if the sulphuric acid is over 3 gms. per 100 c.c.

Second.—The temperature coefficient of mixtures of equal parts copper sulphate and sulphuric acid appears to be that of the sulphuric acid alone.

Third.—The probable error of these determinations is less than 0.5 percent.

Electrochemical Engineering Laboratory,
Pennsylvania State College,
State College, Pa., September 2, 1911.

A paper presented at the Twentieth General Meeting of the American Electrochemical Society, in Toronto, Canada, September 21, 1911, President W. R. Whitney in the Chair.

NEW DATA ON SOME ELECTRICAL PROPERTIES OF SODIUM, POTASSIUM AND THEIR ALLOY.

By Edwin F. Northrup.

In proportion as we ascend from room temperatures, when applying science to industrial processes, there is less and less precise data respecting the physical properties of bodies upon which to place reliance. This is noticeably true in regard to the electrical properties of the pure metals and their alloys at the high temperatures used by the electrochemist. Realizing this, it seemed to the writer well worth while to make an examination of the electrical resistivities and temperature coefficients of as many as possible of the metals and simple alloys industrially used, at temperatures carried as high as possible, and below the melting points of the metals and alloys. That this investigation may be pursued in a systematic manner, it is proposed to take them up in an order based upon the grouping in Mendeléeff's table. The investigation began with sodium and potassium, which fall in Group I, and which do not appear to have been previously examined in respect to resistivities and temperature co-efficients with satisfactory care. As these metals and their alloys have low melting points, there seemed to be no good reason for extending the examination beyond the range of temperature, 0° C. to 150° C. As the electrical properties of all pure metals vary considerably with slight impurities, there would appear to be no advantage, from an engineering standpoint, in attempting great refinements in the measurements of selected samples; because another sample selected with equal care would in this case probably show variations greater than would result from errors in the measurements. The samples examined were the purest which are regularly sold by Merck. The accuracy sought in measuring the temperatures was 1/5° C., and in the resistances 1/5 of 1 percent. The determination of the specific resistances,

upon deductions made from the data, is slightly less than the accuracy of the measurements, for reasons which will appear later.

Some ingenuity had to be exercised to find a convenient method of handling and measuring sodium and potassium because of their peculiar properties and great chemical activity. The methods employed, which will be described in sufficient detail to permit of repetition, were in general as follows: A glass tube, supplied with platinum potential and current terminals, was first filled with pure mercury, and the resistance of this, throughout the range of temperature employed, was determined between potential points. The same tube was then filled with the metal, in a molten state, which was to be measured. Its resistance, between

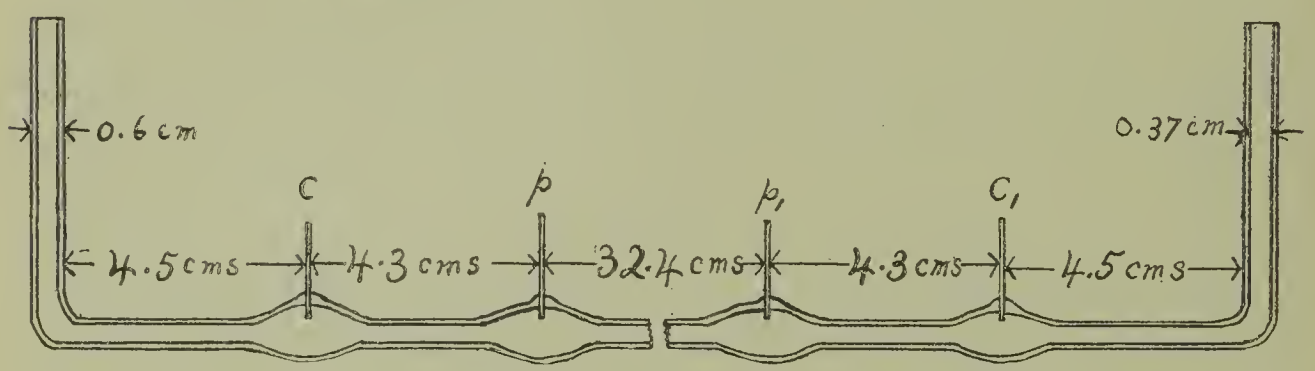


FIG. 1.

the same potential points, was then measured at various temperatures as the metal cooled, from the highest to the lowest temperature. From the data so obtained the specific resistances and temperature coefficients were determined. The resistance measurements were made with a Kelvin Bridge, made by The Leeds and Northrup Company, and known as the Kelvin Bridge, Cat. No. 4305. The glass tube containing the material to be measured was placed in a trough containing paraffin oil, the temperature of which could be varied by an electric heating coil. The temperature measurements were made with a nickel resistance thermometer of approximately the same length as the distance between the potential points in the glass tube, to which the nickel thermometer was attached. Check temperature measurements were occasionally taken with mercury thermometers.

Preparation of the Samples for Measurement.

The glass tube in which the metals to be measured were confined is shown in Fig. 1, with the approximate dimensions used indicated in the figure.

In the method used the resistance values are obtained in terms of pure mercury, measured under the same conditions as the samples. The specific resistance of pure mercury being assumed as accurately known, in the temperature range covered, that of the samples may be deduced as follows.

Let ρ_t be the specific resistance of mercury at temperature t.

Let ρ_t' be the specific resistance of the sample at temperature t.

Let R_t be the measured resistance of the tube filled with mercury at temperature t.

Let R_t' be the measured resistance of tube filled with sample at temperature t.

Let l be the length of tube between potential points.

Let S be the cross-section of the tube.

Then in the case of mercury

$$R_t = \frac{l}{S} \rho_t \qquad (1)$$

and in the case of the sample

$$R_t' = \frac{l}{S} \rho_t' \qquad (2)$$

By taking the ratio of (1) and (2) we obtain

$$\rho_t' = \frac{R_t'}{R_t} \rho_t \qquad (3)$$

which determines the specific resistance of the sample at temperature t, in terms of the specific resistance of mercury at the same temperature. Consequently, the first step in the determinations was to calibrate the tube by filling it with pure mercury and measuring its resistance throughout the range of temperature employed. The mercury was furnished the writer

by Dr. George A. Hulett, and was considered very pure, having been prepared by re-distillations with great care. This was measured in the tube at various temperatures and the results plotted in a curve relating resistance and temperature. The same tube was then filled with molten sodium. But this was not accomplished without considerable experimenting to overcome practical difficulties that arose. The method finally employed, which proved satisfactory, is as follows:

For melting these metals without fire-risk, electric heat was used. A beaker of about one-half liter capacity was wound on the outside with insulated German silver wire, which, together with a rheostat, was attached to a 110-volt circuit. The sodium, under oil, was put in a small beaker and this was placed in the heating beaker, also containing oil. When the sodium melted at a temperature of about 98° C., it was generally found to be in globules covered with a skin which prevented them from being easily united. This difficulty was overcome with both sodium and potassium by allowing them to nearly cool to the point of solidification. The small globules would stiffen first, and then, with a glass rod, could be poked into and made to unite with the larger globules. In this manner the entire mass could be made to unite and be reheated until liquid throughout. To draw the molten metal into the measuring tube, a rubber hose was attached to one end of the measuring tube, and the other end of the rubber hose was attached to a hole in the side of a bottle which was partly filled with oil. Another short glass tube was connected with a short rubber hose to the neck of the bottle. The open end of the measuring tube was then placed in the center of the mass of molten sodium or potassium, and by gentle suction, applied with the mouth at the neck of the bottle, the metal was drawn into the tube in a fluid state, at which it was maintained until cooled down during the progress of the measurements. It was found essential, in order to obtain consistent results to accuracies better than ½ percent, to not allow the metal to freeze in the tube and then reheat before beginning the measurements, the reason of this being that the metal on cooling slightly shrinks away from the walls of the tube, and then on remelting does not completely fill the space occupied at first.

The tube of molten metal so prepared, with the nickel resist-

ance thermometer (consisting of insulated nickel wire wound on a long, thin stick) tied to it, was immersed in the oil bath, where it could be kept at a temperature above the melting point of the metal.

Method of Taking the Observations.

The manner of taking the readings was the same for all the metals measured. The oil was brought with the electric heater to a temperature of about 150° C. The current was then turned off and the observations were taken as the oil cooled down, the oil, of course, being stirred continuously. Two observers were required, one to stir the oil and read on the Kelvin bridge the resistance of the sample, the other to simultaneously read on an accurate Wheatstone's bridge the resistance of the nickel thermometer. This last had been previously calibrated against a standard resistance thermometer and two accurate mercury thermometers. The temperature-resistance curve thus obtained was reliable to within $\frac{1}{5}$° C. When the oil had cooled to room temperature, it was further cooled by drawing some of it off with a siphon and adding cold oil. In the progress of the cooling it was customary to take from 20 to 40 readings. The curves, which were carefully laid out upon cross-section paper 20 x 15 inches, showed consistency and accuracy in the readings. [These curves had to be redrawn for purposes of reproduction.—Ed.] In the Kelvin bridge used, the settings were made by moving a slider which made contact with a rod of manganin of 0.01 ohm resistance. This slider was set before taking each reading for a resistance slightly less than that which would give a balance. When the oil cooled down to a point where the bridge was exactly balanced, as was indicated by the spot of light reflected from the mirror of the D'Arsonval galvanometer used, the resistance of the nickel resistance thermometer was read, which by reference to the curve previously plotted gave the temperature at the instant of balance.

A curious phenomenon developed, which for a time appeared likely to defeat the accuracy of the measurements. Although the platinum potential terminals made contact with the liquid sodium and potassium throughout a length of at least 2 mm., it was discovered that the contact resistance between these platinum terminals and the liquid metal was very variable and averaged

as high as 3 or 4 ohms. Later, however, it was discovered that the momentary passage of a large current through this contact resistance permanently destroyed it, and this was always done before beginning a series of measurements, and the contact resistance was measured at the beginning, middle and end of a series of measurements to make sure that it had not returned.

A set of measurements was first made and then repeated with the tube filled with mercury. Next, more than one set of measurements was made with sodium, then with potassium, and finally with the alloy of sodium and potassium. This alloy was made by heating together equal *volumes* of pure sodium and pure potassium. The manner of doing this was to draw into a 20-c.c.

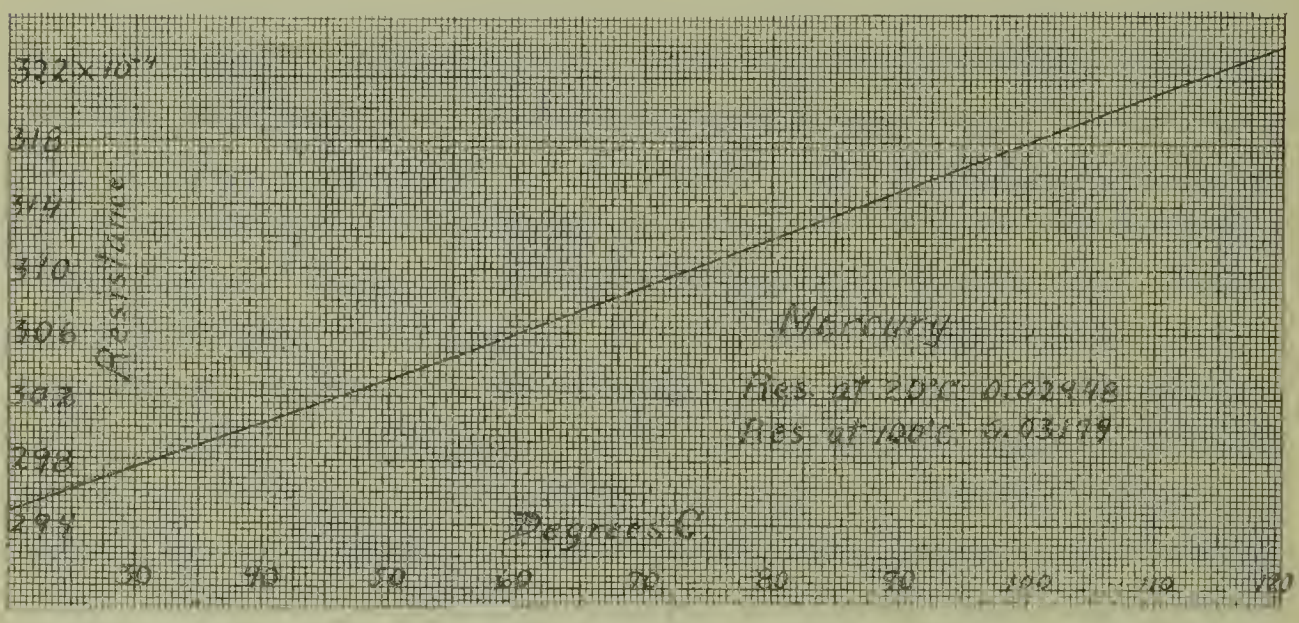


FIG. 2.

pipette a given volume of sodium in the molten state, and then in another like pipette an equal volume of potassium. The two were alloyed together in a small beaker. The alloy so formed is perfectly liquid at room temperatures. When a quantity of it is placed in a tube from which the air has been exhausted, it cannot be distinguished from mercury except by its smaller specific gravity, which is less than 1. Left exposed to the air in a beaker, it becomes instantly covered with a film, and if preserved under kerosene oil or benzine a film also forms, probably due to impurities in the liquid.

A minor object of the investigation was to determine if, perchance, the specific resistance of the alloy would prove to be the

mean of the specific resistance of its components taken in equal parts by volume. Such, however, was by no means the case.

Results Obtained.

Table I gives the values of the resistance of mercury corresponding to the different temperatures measured, which values are plotted in Fig. 2. In column one is also given the resistances corresponding to the different temperatures of the nickel thermometer used.

TABLE I.

Resistance, ohms, of nickel thermometer	Temperature degrees C.	Resistance, ohms, of mercury
133.00	106.8	.03200
131.09	101.8	.03185
128.00	93.8	.03160
117.61	67.2	.03080
111.90	52.5	.03037
111.60	51.6	.03035
100.89	21.5	.02953
100.85	21.48	.02953

The values in Table I denote a relation betwen temperature and resistance for mercury which is sufficiently near linear so that we may calculate values for the resistance of the mercury, for temperatures a few degrees above and below those obtained by experiment, by the formula

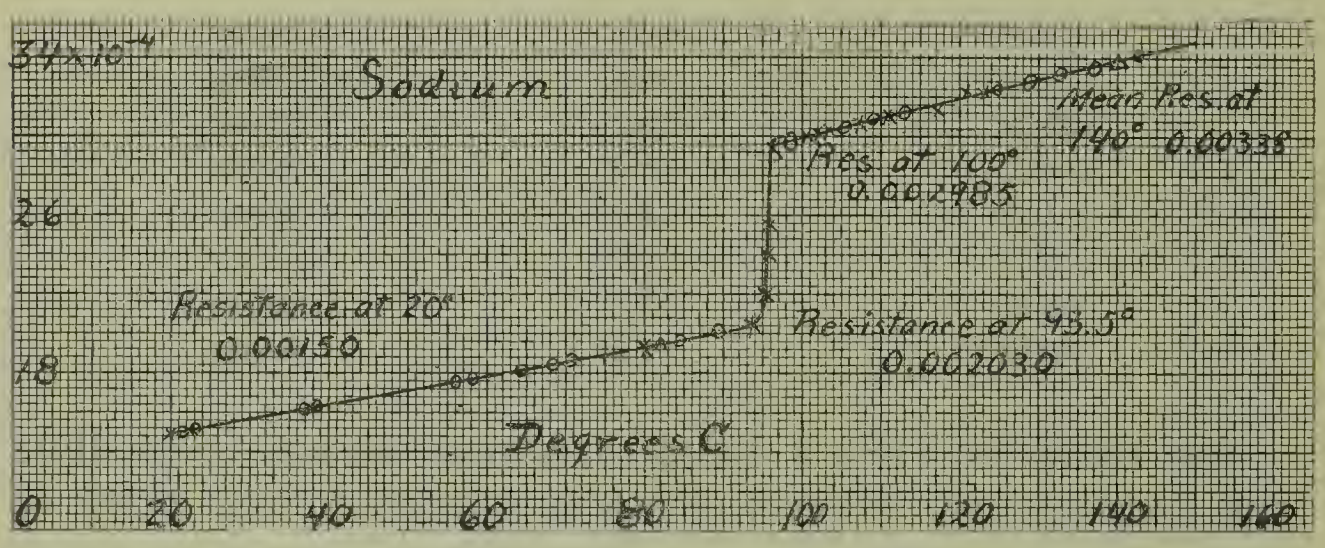
$$R_t = .02889\ (1 + .0010036t) \qquad (4)$$

where t is degrees C. above zero.

It should be noted that when a liquid metal is confined in a glass tube and its resistance is measured between fixed potential points, the values obtained for the temperature coefficient of the metal will be influenced somewhat both by the expansion of the glass and the longitudinal expansion of the metal in the glass. Though these facts are here clearly recognized, we have not deemed it advisable for the purpose in hand to go into a consideration of these refinements. To do so would lead to lengthy physical considerations, whereas the object of this investigation

is to obtain data sufficiently precise for practical and engineering purposes.

In measuring the resistance of sodium it was found, as might be expected, that when the sodium changed from the solid to the liquid state there was an abrupt change in its resistance. The same proved to be true of potassium, and to a less degree of the alloy of sodium and potassium. To study the character of these sudden changes in resistance, a large number of observations were taken, but it is only feasible to record here some sample sets of readings which are thought to represent the facts most accurately. The accuracy of the readings is far greater than can be expressed in curves drawn to a scale that it is prac-



x May 3; o April 28. FIG. 3.

tical to print. The curves given, therefore, are intended to show the character of the variations in the resistances rather than to serve the purpose of accurately expressing values.

Numerical values, however, are marked upon the curves for a few important points, these values being the most probably true values which it is thought the observations give.

Table II gives the observations upon two samples of sodium measured in the same tube, one set of observations being made April 28th, and one set May 3d. On Fig. 3, which is here reproduced, are located the points obtained in these two sets of observations taken at two different times. The accuracy with which the points lie on the curve proves the similarity of the conditions under which the two sets of observations were taken.

TABLE II.

Resistance of Sodium.

Degrees C.	Res. of Na taken April 28th	Res. of Na taken May 3d
21.4	.001511	
22.5	.001515	
35.7	.001605	
36.7	.001610	
55.0	.001740	
58.2	.001760	
64.6	.001810	
68.4	.001840	
70.6	.001860	
80.9	.001940	
83.6	.001960	
89.4	.002000	
93.5		.002030
95.8		.002200
96.3		.002940
97.4		.002950
97.8	.002970	
98.0		.002960
99.0		.002970
100.0		.002980
101.0		.002990
102.0		.003000
102.8		.003001
103.2		.003010
104.0		.003020
104.9	.003040	
107.3		.003050
109.8	.003087	
113.3	.003123	
113.3		.003110
117.3	.003162	
117.3		.003150
120.3		.003180
122.6		.003200
124.0		.003220
128.2	.003269	
132.6	.003312	
136.6	.003349	
141.2	.003394	

Several sets of observations were taken upon both sodium and potassium of the change in resistance as the material was cooled from a temperature slightly above the melting point to a temperature slightly below the melting point, and then as the temperature was again raised from the temperature below the melting point to a temperature above the melting point. The curves so obtained show a loop which is undoubtedly due to a

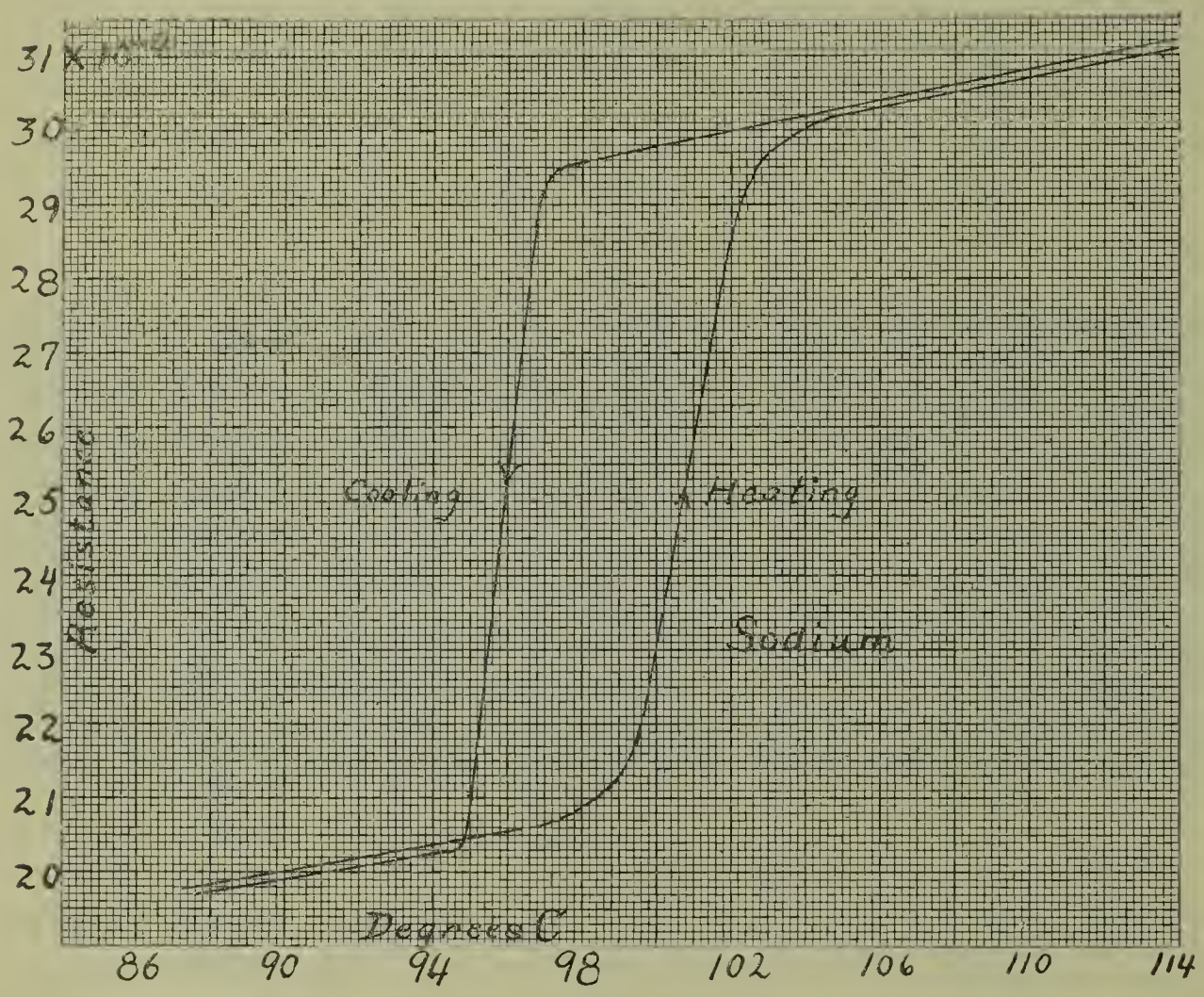


FIG. 4.

lag in the change of state of the material behind the temperature of the oil bath.

Fig. 4 is characteristic of the results obtained by this process. The fact that the cooling curve and the heating curve do not exactly match at a temperature above and below the melting point is explained as follows: When the sodium solidifies it shrinks considerably, and when remelted the liquid occupies a slightly different volume between the potential points in the tube than it did when first put in the tube. This expansion and con-

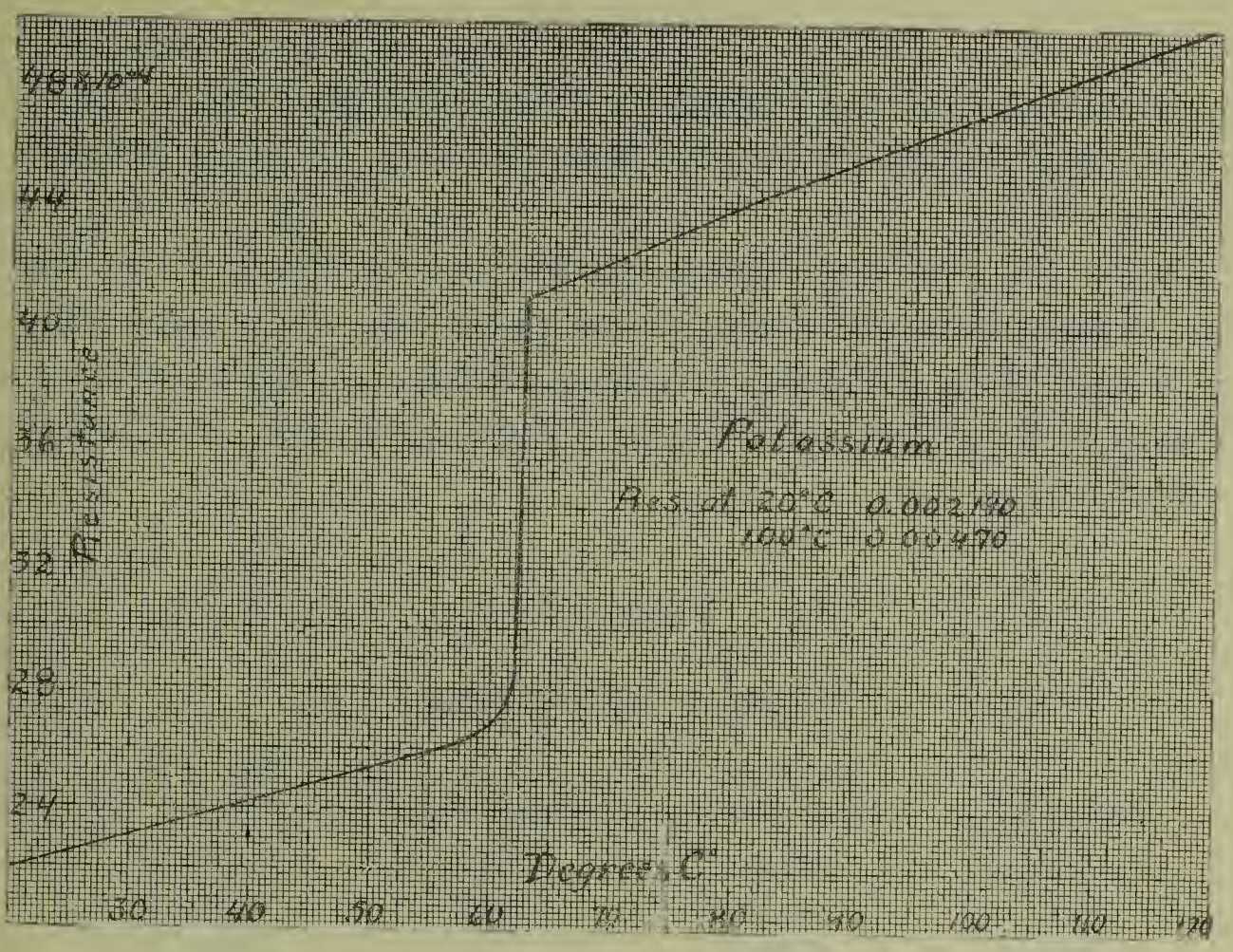

Potassium
Res. at 20°C 0.002120
100°C 0.005470
Degrees C.

FIG. 5.

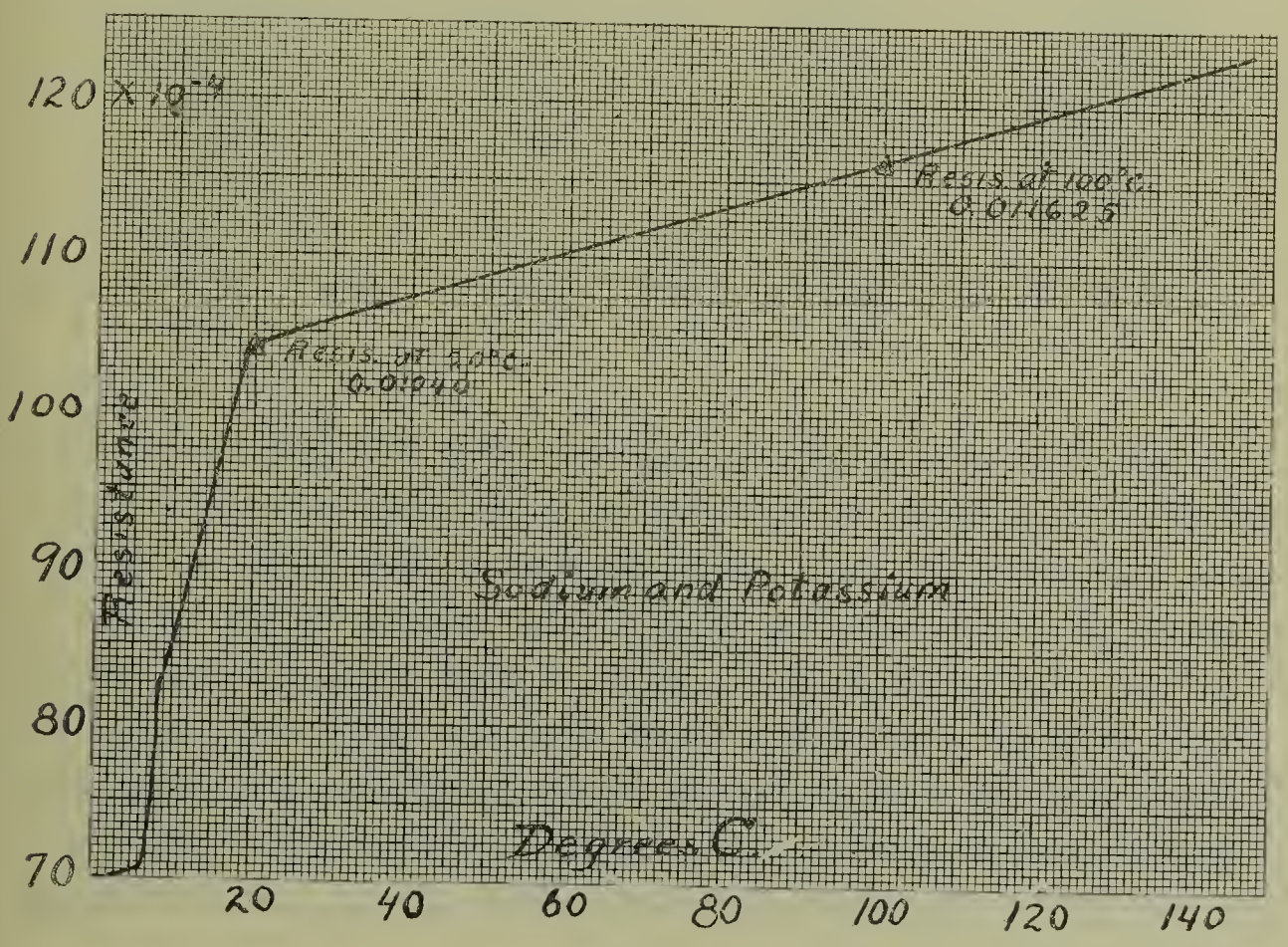

Resistance
Sodium and Potassium
Res. at 20°C. 0.010410
Res. at 100°C. 0.011625
Degrees C.

FIG. 6.

traction of the material is the chief cause why these resistance measurements are not susceptible of the high precision which is easily obtained in measuring the resistance of solid metallic conductors.

Table III and Fig. 5 express the results obtained for the measurement of the resistance of potassium. The same tube was used as in the previous measurements, and great care was exercised to always clean the tube thoroughly each time before refilling.

TABLE III.

Resistance of Potassium.

Degrees C.	Res. of K, taken May 5th	Degrees C.	Res. of K, taken May 5th
21.2	.002204	65.8	.004150
49.9	.002530	68.6	.004200
50.5	.002540	71.0	.004240
55.6	.002600	77.8	.004350
58.7	.002650	81.0	.004410
60.0	.002680	96.6	.004650
60.6	.002700	100.0	.004700
61.4	.002730	103.2	.004750
61.8	.002800	105.7	.004790
62.2	.003100	112.8	.004900
62.4	.003450	116.8	.004960
62.6	.003850	119.3	.005000
63.2	.004100	120.3	.005010

It might be reasonable to suppose, since the alloy of sodium and potassium, taken in equal volumes, is liquid above and below room temperatures, that the resistance of this alloy at a temperature above the melting point of the sodium would prove to be a mean of the resistances of its constituents. It was partly with a view of testing this point that the alloy was measured in the same way and under the same conditions as sodium and potassium. The results show that the above supposition is entirely incorrect. The results obtained for the alloy are presented in Table IV and Fig. 6.

TABLE IV.

Resistance Na + K equal parts by volume.

Degrees C.	Res. Na + K Sample I.	Res. Na + K Sample II.
2.8		.00700
3.4		.00700
4.0		.00702
6.0		.00705
6.6		.00716
6.8		.00724
7.0		.00747
7.4		.00753
7.7		.00784
8.0		.00810
8.3		.00820
8.7		.00825
9.6		.00840
10.3		.00855
11.7		.00880
12.2		.00890
12.5		.00900
13.4		.00920
14.4		.00940
15.2		.00960
16.1		.00980
17.0		.01000
23.4		.01050
37.3		.01070
44.2		.01080
46.1	.01082	
47.4	.01085	
51.6	.01090	
62.2	.01105	
71.0	.01120	
91.3	.01150	
98.2	.01160	
105.0	.01170	
111.8	.01180	
125.0	.01200	
131.2	.01210	

After the above data were taken, in order to make sure that no gross error was being committed, another determination was made of the specific resistance of potassium, use being made of a

tube of entirely different dimensions than the one used in getting the above data. In this new tube the distance between the potential points was 26 cms. and the external diameter of the tube was 3/10 cm. The internal diameter was not measured. This tube was calibrated with mercury. It was then filled with potassium and the resistance of this measured. After allowing the potassium to cool, it was found on reheating it that there was a slight progressive change going on in the resistance, to increase it. The cause of this was not traced, and experiments with this tube were abandoned after a few runs. The results, however, gave the specific resistance of potassium in the first run 3/5 of 1 percent lower and in the second run 3/5 of 1 percent higher than was obtained with the first tube. From this it was concluded that so close an agreement in the results obtained with two tubes of entirely different dimensions insured the correctness of the method. The results relied upon, however, are those which were taken with the first tube, and which are given above.

Discussion of Data.

If a material changes in resistance as a linear function of the temperature, and the resistance is known at two temperatures other than 0°, the temperature coefficient of the material may be calculated as follows:

Let R_o = resistance of material at 0°.

Let R_a = resistance of material at a°.

Let R_b = resistance of material at b°.

Let β = temperature coefficient.

then, $R_a = R_o (1 + \beta a)$

$R_b = R_o (1 + \beta b)$

From these two equations R_o may be eliminated and we derive,

$$\beta = \frac{R_b - R_a}{R_a\, b - R_b\, a}$$

The temperature coefficient derived in this manner assumes that the curve representing the increase in resistance with temperature is a straight line drawn through the two observed temperature points and continued to the temperature 0°. It does

not assume that the coefficient may be used beyond the range over which it was actually determined, which range should always be stated when the coefficient is given. By stating the coefficients of different materials, or of the same material in different temperature ranges, in this way, it is easy to make comparisons of their relative magnitudes. Curves drawn from the data on a large scale show that the resistance changes of sodium and potassium are not strictly linear below the melting points. They are very nearly so above their melting points. To what extent the departure of the curves from a straight line may be due to a contraction of the materials within the glass tubes in which they were measured could only be determined by either an analysis or careful experiments, which the writer has not undertaken. The magnitude of the coefficients is undoubtedly effected, also, by contraction of the material within the tube, to a considerable extent. The coefficients, however, which have been derived under the limitations stated are sufficiently exact for most purposes.

In determining the specific resistances of sodium and potassium and the alloy, the two temperatures, 20° and 100°, have been selected. In making the calculations, values had to be taken for the specific resistance of mercury at each of these temperatures. These values were gotten from Landolt and Börnstein, and are probably considerably more exact than the data obtained upon sodium and potassium and their alloy. The values used are:

$$\text{Specific resistance at } 20° = 95810 \times 10^{-9}$$
$$\text{Specific resistance at } 100° = 103350 \times 10^{-9}$$

With these values as a basis, the specific resistance of sodium, potassium and their alloy have been calculated by formula (3). In the table below are contained all the essential results of the measurements. There is added to this table, for the sake of comparison, equivalent data in regard to copper, and a column of densities. Under the headings, "temperature coefficient," the range over which the coefficient applies without exterpolation beyond the range of the observations is given in brackets.

In the lower part of the table the specific resistance of sodium, potassium and their alloy are also given in terms of mercury, so that if better values for the specific resistance of mercury than

those used above should be obtained, the specific resistances of the materials measured can be easily recalculated, using the new values for mercury.

Substance	Spec. Res. at 20° C. $10^{-9}\times$	Spec. Res. at 100° $10^{-9}\times$	Temp. Coef. Low Range	Temp Coef. High Range	Density at 0°
Mercury . . .	95810	103350	0.001003 (20° to 100°)	. . . . .	13.595
Sodium . . .	4875	9705	0.0053 + (20° to 80°)	0.00494 + (100° to 140°)	0.978
Potassium . .	7118	15275	0.0058 (20° to 50°)	0.00507 + (66° to 120°)	0.87
Alloy, K + Na	33800	37795	. . . . .	0.00147 (46° to 125°)	0.92
Copper . . .	1721	2266	0.00387 +	. . . . .	8.89

Spec. res. of Na			at 20° C.	= 0.05088 ×	Spec. res. of Hg	at 20° C.
" " " Na			at 100° C.	= 0.09390 ×	" " " "	" 100° C.
" " " K			at 20° C.	= 0.07429 ×	" " " "	" 20° C.
" " " K			at 100° C.	= 0.1478 ×	" " " "	" 100° C.
" " " Na+K			at 20° C.	= 0.3528 ×	" " " "	" 20° C.
" " " Na+K			at 100° C.	= 0.3657 ×	" " " "	" 100° C.

Commenting on this table, we would call attention, first, to the very high coefficient of potassium in the range 20° to 50° of 0.0058 and of the slightly smaller coefficient of sodium in the range 20° to 80° of 0.0053. Potassium is thus probably unique in possessing the highest coefficient of any pure metal in this range that has been observed. It should be noted that the coefficient of the alloy of potassium and sodium, instead of being approximately the mean of the coefficients of sodium and potassium when liquid, is only between a third and a fourth of the mean value of the coefficients of its constituents in the same temperature range.

The mean specific resistance at 100° of sodium and potassium is 12490×10^{-9}, while the specific resistance of the alloy, in equal proportions by volume, is 37795×10^{-9}, or slightly over three times as great as the mean specific resistance of its constituents. This is an interesting physical fact which may help to throw light upon the theory of conduction in metals. In this connection we note that alloying metals generally, if not always, increases their specific resistance and lowers their melting point.

The abrupt increase in resistance of the metals experimented with, expressed in percentage, is as follows:

Sodium, in passing from the solid state at 93.5° to the liquid state at 96.3°, increased 44.8 percent.

Potassium, in passing from the solid state at 60.4° to the liquid state at 63.2°, increased 52.2 percent.

The alloy of sodium and potassium, in passing from 2.8°, at which temperature it may probably be considered as completely solid, to 20° C., at which temperature it may probably be considered as completely liquid, increased in resistance 48.5 percent.

In order that these abrupt changes in resistance may be used to find the true melting point of the metals it would be necessary to plot the temperature-resistance curve as obtained with a falling temperature, and again as obtained with a rising temperature, and then take as the melting temperature of the metal the mean value of the resistance when changing abruptly in the two cases. Thus, referring to Fig. 4, we would determine the true melting point of sodium on this basis to be 98.4°. In another, rather rough, determination of the change in resistance of potassium made in a smaller tube (the results of which measurements are not recorded here) the melting point was found to be, on the same basis as above, 63.5°. These values are slightly higher than those usually given for these metals, but this may be due to the supposed high purity of the metals experimented with. If this method were to be used for determining melting points, the metals should be confined in thin-walled capillary tubes to reduce the temperature lag to a minimum.

The property exhibited by sodium, potassium and their alloy of abruptly increasing in resistance in passing through a change of state is probably not confined to these metals, and may belong to all pure metals and many alloys. If such be the case, one would fall into grave errors in attempting to determine resistivities of metals above their melting points from data obtained respecting these metals below their melting points. For example, the ring of molten iron in an induction furnace probably has a much higher resistance than would be obtained by calculation from the temperature-resistance curve of iron which ends below its melting point.

The ratio of the resistivity at 20° C. of sodium to copper is 3.058, but the density of copper is almost exactly nine times that of sodium, which, in round numbers, makes sodium three times as good a conductor per unit of weight as copper. Hence it could compete with copper as a conveyor of electric power if it cost anything less than 3 times as much as copper per pound, provided, of course, current-carrying capacity were the only consideration. It has been suggested before now that sodium might be used in iron pipes in a practical way for carrying electric power. The limits of this article will not permit the writer to discuss the feasibility of this proposal, and it is here merely suggested that, inasmuch as electrochemical processes often necessitate the carrying over short distances of enormous currents, the consideration of the proposal might be with advantage taken up afresh.

I have found that the work of others on the change in resistance of metals confirms these statements, although not very much work, that I can find, has been done along this line.

As a matter of fact, I have quotations on the price of sodium in carload lots—25 cents a pound. I think the gain would be perhaps something like 33 percent. In a number of places, very large currents are conveyed comparatively short distances, 500 feet or so, and it really would seem practicable to lay a pipe line and fill that pipe line with metallic sodium. It is hoped to continue this investigation to very high temperatures with a number of metals.

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The resistivities of sodium and potassium have been determined by other observers. The following values, expressed in a way to permit of easy comparison with the writer's, are copied from Landolt and Börnstein.

Spec. res. of	Na at	21.7° C.	= 4464×10-9.....	Matthiessen
" " "	Na at	120.2° C.	= 11325×10-9.....	"
" " "	Na at	0.0° C.	= 4739×10-9.....	Bernini
" " "	Na at	120° C.	= 8756×10-9.....	"
" " "	K at	0° C.	= 6644×10-9.....	"
" " "	K at	100° C.	= 16501×10-9.....	"

Since the above was written, the writer received, in reply to a request made by him to the National Bureau of Standards at

Washington, the following statements in regard to the resistivities of pure mercury:

"We are pleased to give you, ..., the values computed from the temperature formulas of F. E. Smith, of the National Physical Laboratory of England (Phil. Trans. 204, p. 112; 1904), C. E. Guillaume, of the International Bureau (Comptes Rendus, 115, p. 414; 1892), and Kreichgauer and Jaeger, of the Reichsanstalt (Wied. Ann., 47, 527; 1892). In the following table, volume resistivity is given in microhms per centimeter cube:

	Volume Resistivity		
	0°C	20°C	100°C
Smith	94.073	95.783	103.410
Gillaume	94.073	95.782	103.379
Kreichgauer and Jaeger	94.073	95.782	103.56

* * * * * * * * *"

Adopting the values of Kreichgauer and Jaeger of 95782×10^{-9} and 103560×10^{-9} as the resistivities of mercury at 20° and 100° C., respectively, we obtain the following values of the resistivities of sodium, potassium and their alloy:

Spec. res. of Na	at 20°	=	4873×10^{-9}
Spec. res. of Na	at 100°	=	9724×10^{-9}
Spec. res. of K	at 20°	=	7116×10^{-9}
Spec. res. of K	at 100°	=	15306×10^{-9}
Spec. res. of Na+K	at 20°	=	33792×10^{-9}
Spec. res. of Na+K	at 100°	=	37872×10^{-9}

Palmer Physical Laboratory,
Princeton, New Jersey,
June, 1911.

DISCUSSION.

MR. W. L. MORRISON: In Fig. 4 I notice that the cooling of the sodium changes the resistance. I would like to know if the author heated that sodium, or reheated it, in the same tube, because there might have been sodium silicate formed upon the glass tube, which would account for the change in the resistivity on cooling.

Dr. E. F. Northrup: All this work was done in the same tube at all times, and it was cooled and heated repeatedly, and a large number of these loops were obtained which lay very closely over one another, but I never could get them to match exactly at the bottom and at the top. It is probably due to shrinkages on solidification.

I might say that you can take these results on page 203 as accurate to one-third of one percent, hardly better than that; but you can rely on them to that accuracy.

President Whitney: In connection with what Mr. Morrison said, it is well known that a number of other metals, bismuth, for example, greatly increase in resistivity, and others, as cadmium, decrease in resistivity, as they pass through the melting point.

Mr. Carl Hering: Does anyone here happen to know whether iron or steel is subject to such great changes at its melting point, and approximately what they are quantitatively? From some crude experiments I made, it seems there must be a similar rise in resistivity for steel, and very probably it is considerably greater than that for these alkaline metals.

Dr. Northrup: I saw an article in *The Philosophical Magazine* which referred to the work of Vassura, who found the ratio of the resistivity just after fusion to that just before to be, for tin, 2.1, and for cadmium, 2.3. De la Rive has given the corresponding ratios for two other metals, zinc, 2.0, and lead, 1.9. I shall expect, when I try other materials, to find like changes in resistivity as fusion takes place.

Dr. M. G. Lloyd: In discussing the subject of conductivity and temperature coefficient, it might be interesting to mention the work at very low temperatures recently done at Leyden by Onnes, making use of liquid helium. He has found that the pure metals approach as closely to zero resistance as he is able to measure. That is to say, the resistance goes down to something like 0.0000001 part of the resistance at ordinary temperatures, so low that within the accuracy of measurement it can be called zero. That was found not only with mercury, but with gold, platinum and some other metals. On the other hand, the alloys do not show any such continual decrease as absolute zero is approached.

A paper presented at the Twentieth General Meeting of the American Electrochemical Society, in Toronto, Canada, September 21, 1911, President W. R. Whitney in the Chair.

THE ELECTRICAL RESISTANCE OF IRON ALLOYS.*

By Charles F. Burgess and James Aston.

In the design of electrical machinery, the electrical resistance of the iron used for electromagnets is of an importance second only to the magnetic permeability, particularly for alternating current working. A high resistance is desired, since it will reduce to a minimum the eddy currents set up by the reversals of the magnetic flux. It is also of interest to know the conductivity of the various iron alloys, regardless of their magnetic properties, because of their possible utility in rheostats, resistance heating wires and the like.

We therefore, in connection with a series of tests on the various properties of electrolytic iron and its alloys, determined the electrical resistance of the numerous bars used in the magnetic tests. Starting with electrolytic iron as a base material, and making up the alloys with every precaution to avoid contamination, the results given represent the influence of the addition elements.

It is to be regretted that many of the compositions given are based upon the added amounts of the several elements, and have not been checked by analysis. However, the compositions noted for upwards of eighty of the binary alloys are those determined by analysis; and the elements used in the more complex series are largely those which readily alloy with iron in the added proportions. The analyzed bars can be readily recognized in the tables, since the compositions are given to the third decimal place. Again, the curves of Figs. 1 and 2 are plotted from these accurately determined compositions.

Since all of the bars were of a uniform diameter of one centimeter, a measurement of the drop of potential over a length of 7.854 cm. is equivalent to that taken over 10 cm. length of a bar

* This investigation is being conducted in the Chemical Engineering laboratories of the University of Wisconsin largely through a grant of the Carnegie Institution of Washington.

1 cm. square. The apparatus used consisted of two knife edges, 7.854 cm. apart, upon which the test bar was laid, and pressed into firm contact by means of spring clips; the latter were outside the knife edges, and served also as conductors to lead the current

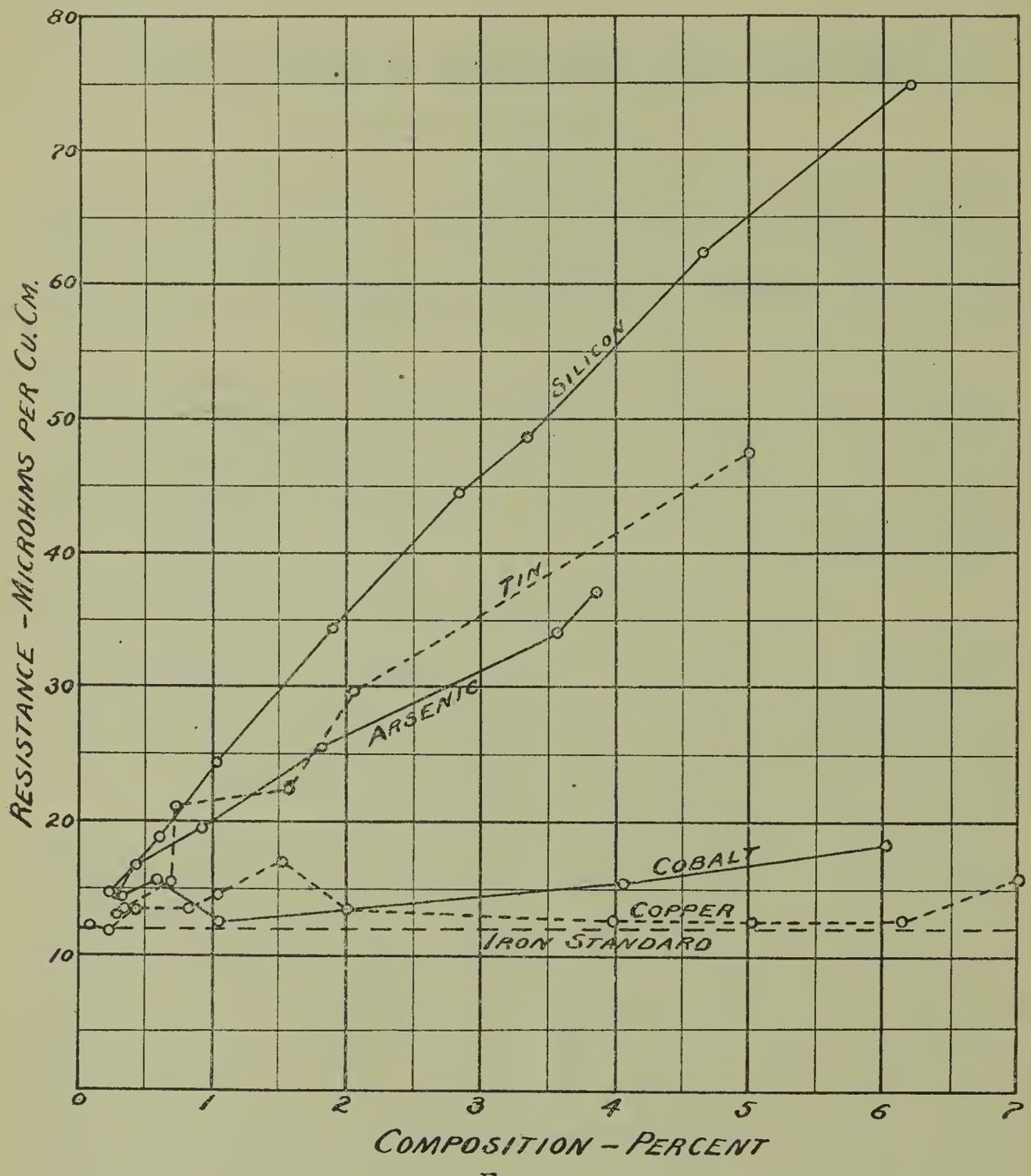

FIG. 1.

into the bar. The current was measured by an ammeter, and the fall of potential by a millivoltmeter. Such values of current were used, up to a maximum of 50 amperes, as would give a large deflection on the millivoltmeter scale, a 0-20 instrument. From these two readings a simple slide-rule calculation gave the specific

resistance in terms of Bar 117 A, of forged electrolytic iron. This bar has a resistance of 12.1 microhms per cubic centimeter in the quenched condition, a value which is not the minimum observed for electrolytic iron. But it was thought best to adhere to the same standard used for the magnetic tests data.

The results of these measurements are noted in Table I, Figs.

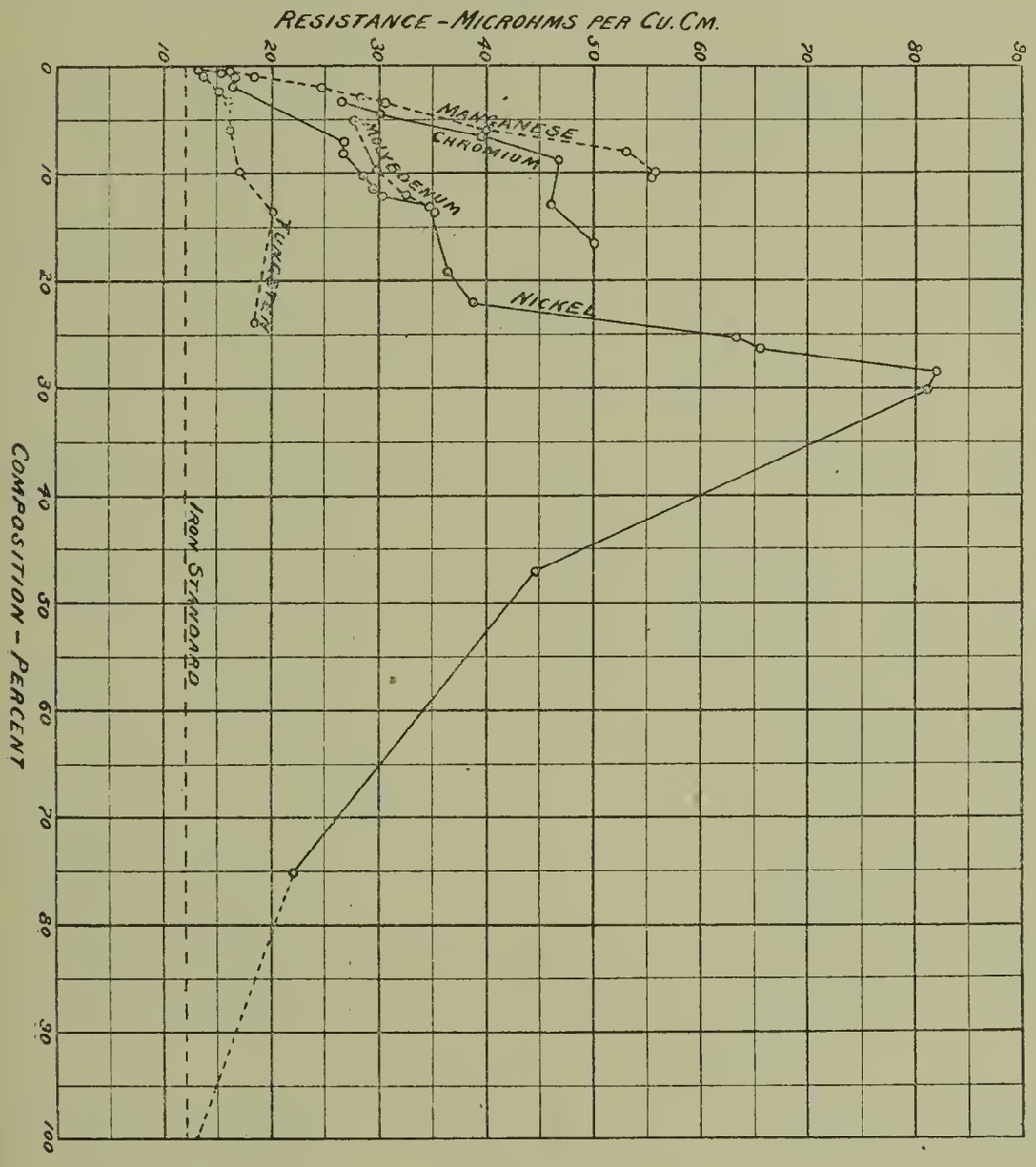

FIG. 2.

1 and 2. The arrangement is in groupings in alphabetical order under the headings of the alloying element, in the binary series; ternary or higher alloys appear in the group of the principal alloying element. In the final miscellaneous group appear those materials in which are more than one of the five elements—chromium, manganese, molybdenum, nickel and tungsten, which tend to produce self-hardening in steels.

TABLE I.

Electrical Resistance.

No.	Bar	Composition	As Forged		After Treatment		
			Mohs. per Cm.^{3}	Rela. Res.	Mohs. per Cm.^{3}	Rela. Res.	Condition
		IRON					
1	NSW	Swedish Anode			11.6	0.96	Quench 900
2	117A	Electro Forged			12.1	1.00	" "
3	AB	" "			11.1	0.92	" "
4	ELI	" "			11.1	0.92	" "
5	172A	" "	14.0	1.16			
6	117B	" "	13.1	1.08	10.6	0.88	Anneal 1000
7	117C	" "	13.4	1.11	13.4	1.11	" "
8	AC	" "	10.7	0.88	10.9	0.90	" "
9	85	" "	14.4	1.19	13.0	1.07	" "
		ALUMINUM					
10	55D	0.067 Al			23.9	1.98	Quench 900
11	76C	0.8 Al	20.6	1.70	16.1	1.33	Anneal 1000
12	109C	1.333 Al			17.4	1.44	Quench 900
		ANTIMONY					
13	172E	1.0 Sb			20.2	1.67	Anneal 1000
		ARSENIC					
14	121I	0.292 As			14.7	1.22	Quench 900
15	121H	0.430 As			16.8	1.39	" "
16	113A	0.915 As			19.5	1.62	" "
17	92A	1 As	18.1	1.50	14.3	1.18	Anneal 1000
18	113B	1.810 As			25.5	2.11	Quench 900
19	113C	3 As	25.3	2.09	26.1	2.15	Anneal 1000
20	113D	3.862 As			37.1	3.07	Quench 900
21	113H	3.563 As			34.0	3.82	" "
22	113E	5 As	26.7	2.21	20.8	7.72	Anneal 1000
23	172C	Transformer			18.7	1.55	" "
24	172D	Transformer + 5 As			30.2	2.51	" "
		BISMUTH					
25	172G	1.0 Bi			13.4	1.11	Anneal 1000
26	172H	2.0 Bi			14.5	1.20	" "
27	172J	4.0 Bi			15.3	1.27	" "
28	172K	6.0 Bi			13.0	1.08	" "
29	172L	10.0 Bi			16.6	1.38	" "
		CHROMIUM					
30	137C	3.27 Cr			26.7	2.21	Quench 900
31	133E	4 Cr 0.84 Si	41.6	3.44	46.9	3.88	Quench 1000
32	133D	5 Cr 0.84 Si	39.6	3.27	39.6	3.27	" "
33	133B	5 Cr 1.4 Si	47.7	3.94	57.0	4.72	" "
34	160J	5 Cr 0.3V	35.0	2.89	35.4	2.93	" "
35	160H	5 Cr 0.5V	34.4	2.84	34.4	2.84	" "
36	160K	5 Cr 1 V	36.1	2.98	26.8	2.22	" "
37	133L	6 Cr 0.4 Si	35.0	2.89	42.4	3.50	" "

TABLE I—*Continued.*
Electrical Resistance.

No.	Bar	Composition	As Forged		After Treatment		
			Mohs. per Cm.^{3}	Rela. Res.	Mohs. per Cm.^{3}	Rela. Res.	Condition
		CHROMIUM—Cont.					
38	133K	6 Cr 0.84 Si	42.4	3.42	49.1	4.06	Quench 1000
39	147Z	6 Cr 1.3 Si	47.4	3.92	59.5	4.92	" "
40	133F	6.00 Cr.	52.5	4.34	64.4	5.32	" "
41	137E	6.68 Cr			39.7	3.28	Quench 900
42	133M	8 Cr 1 Si	53.2	4.29	61.2	5.06	Quench 1000
43	78C	8.7 Cr 1 Cu 1 Al	35.6	2.94			
44	121G	8.83 Cr			46.8	3.87	Quench 900
45	111S	10 Cr 2 Si	75.0	6.20	59.8	4.94	Quench 1000
46	155K	10 Cr 2 Si 0.3 V 0.6 C	59.2	4.89	77.4	6.40	" "
47	137L	10 Cr 2 Si 2 Ti	79.5	6.57	86.0	7.11	" "
48	122E	10 Cr 3 Si	87.5	7.23	84.0	6.95	" "
49	112A	10 Cr 5 Si	103.0	8.50			
50	160I	10 Cr 0.3 V	49.2	4.07	45.7	3.78	" "
51	155L	10 Cr 0.3 V 0.65 C	49.9	4.13	55.0	4.55	" "
52	160G	10 Cr 0.5 V	51.3	4.24			
53	163O	10 Cr 1 V	45.9	3.79	74.8	6.18	" "
54	141C	10.3 Cr 1 V 2 Si 0.3 C	83.4	6.89	84.9	7.01	" "
55	136G	13.01 Cr			46.0	3.80	Quench 900
56	133N	13 Cr 1.8 Si	75.8	6.26	85.6	7.08	Quench 1000
57	141E	15 Cr 2 Si	92.6	7.55			
58	137H	16.57 Cr.			50.0	4.13	Quench 900
59	136H	17 Cr 1½ Si	72.8	6.02	76.0	6.28	Quench 1000
		COBALT					
60	123A	0.32 Co			14.4	1.19	Quench 900
61	123B	0.580 Co			15.5	1.28	" "
62	123C	1.035 Co			12.5	1.03	" "
63	123D	2.000 Co			13.5	1.12	" "
64	123E	3.0 Co	14.7	1.21	11.7	0.97	Quench 1000
65	123F	4.055 Co			15.4	1.27	Quench 900
66	123G	6.020 Co			18.3	1.51	" "
		COPPER					
67	147B	0.089 Cu			12.2	1.01	Quench 900
68	158A	0.202 Cu			12.0	0.99	" "
69	158B	0.422 Cu			13.6	1.12	" "
70	86A	½ Cu	14.0	1.16	12.2	1.01	Anneal 1000
71	92F	½ Cu ¼ Si	17.0	1.40	13.1	1.08	" "
72	90F	½ Cu ½ Si	22.0	1.82	21.8	1.80	" "
73	158D	0.804 Cu			13.6	1.12	Quench 900
74	113T	1 Cu	11.4	0.93	11.8	0.96	Anneal 1000
75	50A	1 Cu	16.4	1.36	13.2	1.09	" "
76	92C	1 Cu ½ Si	21.7	1.79	18.3	1.51	" "
77	90E	1 Cu 1 Si	22.9	1.89	19.2	1.59	" "
78	103G	1 Cu 1 Sn	16.7	1.40			" "
79	111U	1 Cu ½ Al	18.8	1.55	14.5	1.20	" "
80	147H	1.006 Cu			14.7	1.22	Quench 900

TABLE I—*Continued.*
Electrical Resistance.

No.	Bar	Composition	As Forged		After Treatment		
			Mohs. per Cm.^{3}	Rela. Res.	Mohs. per Cm.^{3}	Rela. Res.	Condition
		COPPER—Continued					
81	147J	1.510 Cu			17.0	1.41	Quench 900
82	84B	2 Cu	15.6	1.29	11.2	0.93	Anneal 1000
83	88B	2 Cu 1 Si	24.6	2.03	22.2	1.83	" "
84	144AE	2 Cu ½ Al	22.1	1.63	19.7	1.64	" "
85	111V	2 Cu ½ Al	18.5	1.53			
86	158G	2.005 Cu			13.4	1.11	Quench 900
87	111L	2½ Cu			9.5	0.79	Anneal 1000
88	111M	3 Cu	15.5	1.28	12.4	1.03	" "
89	111R	3 Cu 1 As	22.1	1.83	15.1	1.25	" "
90	94F	3 Cu ½ Si	20.2	1.67	8.1	0.67	" "
91	88C	3 Cu 1 Si	25.3	2.09	17.8	1.47	" "
92	111X	3 Cu 1 Al	21.8	1.80	8.0	0.66	" "
93	113W	3½ Cu	14.7	1.22	8.6	0.71	" "
94	158I	3.990 Cu			12.7	1.05	Quench 900
95	121L	4 Cu	13.6	1.12	12.5	1.03	Anneal 1000
96	86C	5 Cu	15.7	1.30	12.8	1.06	" "
97	121K	5 Cu 1 As	20.5	1.69	19.6	1.62	" "
98	83C	5 Cu 1 Si	25.3	2.09	22.6	1.87	" "
99	88D	5 Cu 2 Si			26.5	2.19	" "
100	144AD	5 Cu 1 Al	20.0	1.65	18.1	1.50	" "
101	111Y	5 Cu 1 Al	19.9	1.64	17.6	1.29	" "
102	158J	5.070 Cu			12.5	1.03	Quench 900
103	113U	6 Cu 1 Si			22.8	1.88	Anneal 1000
104	113S	6 Cu 1 Al	19.8	1.64	17.6	1.45	" "
105	158K	6.160 Cu			12.7	1.05	Quench 900
106	88A	6.67 Cu 6.67 Si	23.4	1.93	15.7	1.30	Anneal 1000
107	147U	7.050 Cu			15.8	1.31	Quench 900
108	86E	90 Cu	5.53	0.46			
109	49A	90 Cu	5.09	0.42			
110	86F	94.340 Cu			3.92	0.24	Quench 900
111	50C	98 Cu	4.46	0.37			
		LEAD					
112	122B	0.061 Pb			13.9	1.15	Quench 900
		MANGANESE					
113	123H	0.505 Mn			16.1	1.33	Quench 900
114	170A	1 Mn			18.4	1.52	Anneal 1000
115	170B	2 Mn			24.8	2.05	" "
116	170C	3 Mn			28.4	2.35	" "
117	144V	3½ Mn	30.2	2.58	30.6	2.53	" "
118	123I	4.514 Mn			30.1	2.49	Quench 900
119	170D	6 Mn	40.8	3.37	40.0	3.31	Quench 1000
120	170E	8 Mn	49.1	4.06	53.0	4.38	Anneal 1000
121	170F	10 Mn	54.8	4.53	55.8	4.61	" "
122	123J	10.419 Mn			55.3	4.57	Quench 900

TABLE I—*Continued.*

Electrical Resistance.

No.	Bar	Composition	As Forged		After Treatment		
			Mohs. per Cm.^{3}	Rela. Res.	Mohs. per Cm.^{3}	Rela. Res.	Condition
		MOLYBDENUM					
123	160E	4 Mo 0.6 V	17.8	1.47	22.6	1.87	Quench 1000
124	163M	5 Mo 1 V	22.9	2.47	25.0	2.06	" "
125	155N	5.12 Mo			27.8	2.30	Quench 900
126	160D	8 Mo 0.5 V	25.1	2.07	25.4	2.10	Quench 1000
127	156E	8 Mo 0.3 V 0.6 C	22.6	1.87	27.8	2.30	" "
128	155M	9.84 Mo			29.9	2.47	Quench 900
129	160A	10 Mo 0.3 V	27.3	2.25	27.2	2.25	Quench 1000
130	160C	10 Mo 0.3 V 0.4 C	17.1	1.41	26.6	2.20	" "
131	160B	10 Mo 0.5 V	30.7	2.54	30.3	2.51	" "
132	163I	10 Mo 1 V	30.1	2.49	29.4	2.47	" "
133	163N	10 Mo 1 V	30.1	2.65	32.1	2.65	" "
134	163H	10 Mo 1 V 0.4 C	24.2	2.00	19.9	1.65	" "
135	141F	10 Mo 1.2 C	25.2	2.08	29.5	2.44	" "
136	146Y	12 Mo	36.6	3.02	32.6	2.70	" "
137	155O	13.69 Mo			35.1	2.90	Quench 900
		NICKEL					
138	144C	0.270 Ni			13.1	1.08	Quench 900
139	144D	0.560 Ni			15.4	1.27	" "
140	144E	1.070 Ni			16.9	1.40	" "
141	144F	1.93 Ni			16.4	1.36	" "
142	160N	5 Ni 0.3 V	22.3	1.84	24.6	2.08	Quench 1000
143	160O	5 Ni 0.6 V	24.5	2.02	25.0	2.06	" "
144	144J	7.05 Ni			26.9	2.22	Quench 900
145	157A	8.17 Ni			26.7	2.20	" "
146	160L	10 Ni 0.3 V	30.1	2.49	29.4	2.47	Quench 1000
147	160M	10 Ni 0.6 V	31.2	2.58	32.2	2.66	" "
148	146P	10 Ni 2 Si 1 C	54.5	4.50	70.3	6.03	" "
149	157D	10.20 Ni			28.6	2.36	Quench 900
150	157E	11.29 Ni			29.4	24.3	" "
151	157H	12.07 Ni			30.3	2.50	" "
152	144M	13.11 Ni			34.8	2.62	" "
153	144P	19.21 Ni			36.2	2.99	" "
154	166G	22.11 Ni			38.7	3.20	" "
155	154S	25.20 Ni			63.2	5.22	" "
156	166I	26.40 Ni			65.5	5.41	" "
157	166C	28.42 Ni			82.0	6.77	" "
158	166L	35.09 Ni			81.1	6.70	" "
159	166O	47.08 Ni			44.7	3.69	" "
160	166Q	75.06 Ni			22.1	1.83	" "
		SELENIUM					
161	121D	0.017 Se			16.2	1.34	Quench 900
162	121E	0.019 Se			14.6	1.21	" "

TABLE I—*Continued.*

Electrical Resistance.

No.	Bar	Composition	As Forged		After Treatment		
			Mohs. per Cm.^{3}	Rela. Res.	Mohs. per Cm.^{3}	Rela. Res.	Condition
		SILICON					
163	96F	0.233 Si			14.7	1.22	Quench 900
164	83A	½ Si	19.5	1.61	19.8	1.64	Anneal 1000
165	96A	0.603 Si			18.7	1.55	Quench 900
166	44A	1 Si	18.6	1.54	15.9	1.31	Anneal 1000
167	96B	1.033 Si			24.3	2.01	Quench 900
168	96C	1.897 Si			34.4	2.84	" "
169	126D	2 Si 10 V	78.9	6.51	75.5	6.24	Anneal 1000
170	109B	2½ Si	45.7	3.78	44.2	3.65	" "
171	109A	2.826 Si			44.6	3.68	Quench 900
172	113V	3.334 Si			48.6	4.02	" "
173	111N	4 Si	54.8	4.53			
174	44D	4 Si	48.6	4.02			
175	96H	4.655 Si			62.2	5.13	Quench 900
176	83B	5 Si	40.3	3.33	36.9	3.05	Anneal 1000
177	109D	6.2 Si	77.2	6.37	74.9	6.18	" "
		SILVER					
178	118A	0.281 Ag			12.5	1.03	Quench 900
179	118B	0.492 Ag			13.3	1.10	" "
180	113K	0.533 Ag			13.1	1.08	" "
181	113L	0.691 Ag			13.8	1.14	" "
182	113M	0.506 Ag			13.6	1.12	" "
183	122A	0.677 Ag			13.5	1.12	" "
184	118C	0.581 Ag			14.7	1.21	" "
185	113N	4 Ag	12.2	1.01	7.7	0.64	Anneal 1000
		TIN					
186	111B	0.288 Sn			13.0	1.07	Quench 900
187	103C	0.342 Sn			13.3	1.10	" "
188	103D	0.686 Sn			15.4	1.27	" "
189	111&	0.717 Sn			21.1	1.74	" "
190	103H	1 Sn 2 Al	18.9	1.56	1.82	1.50	Anneal 1000
191	103E	1Sn 1 Si	28.1	2.32	19.6	1.62	" "
192	111A	1.568 Sn			22.4	1.85	Quench 900
193	121J	2.059 Sn			29.8	2.46	" "
194	111Q	3 Sn 0.23 P	25.6	2.12	25.6	2.12	Anneal 1000
195	113R	3½ Sn	24.1	1.99	25.0	2.06	" "
196	111D	5 Sn			47.4	3.92	" "
		TUNGSTEN					
197	154V	0.406 W			13.4	1.11	Quench 900
198	154W	0.93 W			13.6	1.12	" "
199	154X	2 W	19.8	1.63	17.6	1.45	Anneal 1000
200	154Y	2.33 W			15.1	1.25	Quench 900
201	154AB	3.35 W			16.1	1.33	" "
202	154Z	4 W	19.5	1.61	15.4	1.27	Anneal 1000

TABLE I—*Continued.*

Electrical Resistance.

No.	Bar	Composition	As Forged		After Treatment		
			Mohs. per Cm.^{3}	Rela. Res.	Mohs. per Cm.^{3}	Rela. Res.	Condition
		TUNGSTEN—Continued					
203	160P	4 W 0.4 V	18.1	1.50	20.0	1.65	Quench 1000
204	163J	4 W 0.4 V 0.5 C	23.2	1.92	38.9	3.22	" "
205	163D	4 W 0.4 V 0.6 C	23.0	1.90	35.0	2.89	" "
206	163K	4 W 0.4 V 0.8 C	24.1	1.99	43.1	3.56	" "
207	155X	5 W 0.5 C	18.5	1.53	24.5	2.02	" "
208	155A	5.98 W			16.2	1.34	Quench 900
209	154L	6 W	24.2	2.00	16.1	1.33	Anneal 1000
210	WIC	6 W + C?	22.6	1.87			
211	160Q	7 W 0.3 V	20.3	1.68	22.2	1.84	Quench 1000
212	163E	7 W 0.3 V 0.6 C	24.1	1.99	33.3	2.75	" "
213	155B	8 W	19.9	1.65	18.2	1.50	Anneal 1000
214	155C	9 W	23.0	1.90	16.5	1.36	" "
215	155D	9.85 W			17.1	1.41	Quench 900
216	155E	12 W	20.7	1.71	17.8	1.47	Anneal 1000
217	155F	13.64 W			20.2	1.67	Quench 900
218	155G	16 W	24.2	2.00	19.3	1.59	Anneal 1000
219	155H	18 W	23.4	1.93	18.7	1.54	" "
220	155I	23.87 W			18.5	1.53	Quench 900
		MISCELLANEOUS					
221	136L	20 Cr 3 Mn 1 Si	65.3	5.39	67.1	5.55	Quench 1000
222	141G	21.4 Cr 3 Mn 1 Si 1V	76.8	6.34	82.0	6.78	" "
223	150C	2 Cr 10 MO 0.5 C 0.3 V	37.0	3.06	38.9	3.22	" "
224	155S	10 Cr 5 Mo	47.9	3.96	50.0	4.13	" "
225	155R	10 Cr 10 Mo	48.1	3.98	49.4	4.08	" "
226	155U	2 Cr 10 W	28.0	2.31	28.3	2.34	" "
227	156F	3 Cr 8 W	30.0	2.48	30.5	2.52	" "
228	163A	5 Cr 2 W 0.6 C	47.4	3.92	57.8	4.78	" "
229	163G	5 Cr 5 W 0.6 C	56.8	4.70	57.8	4.78	" "
230	148A	6 Cr 18 W 0.3 V 0.69 C	50.3	4.15	48.0	3.97	" "
231	126B	7.23 Cr 9 W 3 Si	89.5	7.39	87.9	7.26	" "
232	163C	10 Cr 2W	51.4	4.25	51.5	4.26	" "
233	156K	10 Cr 2 W 2 Si	72.8	6.02			
234	156J	10 Cr 2 W 0.6 C	53.6	4.43	55.7	4.60	" "
235	156H	10 Cr 2 W 0.3 V	53.3	4.40	49.8	4.12	" "
236	156I	10 Cr 2 W 0.3 V 0.6 C	60.0	4.95	60.4	5.00	" "
237	126A	10Cr 5 W 3 Si	92.2	7.61	87.9	7.26	" "
238	155V	10 Cr 10 W	60.7	5.02	57.8	4.78	" "
239	150B						
240	150D	2Cr 10W 10Mo 0.3V 0.4C	45.8	3.78	38.3	3.16	Quench 1000
241	163B	2Cr 16W 5Mo 0.3V 0.5C	44.5	3.68	38.8	3.21	" "
242	156G	7 Cr 3 W 2 Mo	43.9	3.63	40.9	3.38	" "
243	156M	10 Cr 2 Mo 2 W	50.9	4.20	49.7	4.11	" "
244	156P	10 Cr 2 W 2 Mo 0.6 C	61.6	5.09			" "
245	136M	10Cr 2W 2Mo 2Si 0.3V	77.4	6.39	83.8	6.93	" "
246	140Q	5 Cr 3 Mn 4 Mo 1 Si	48.6	4.02	54.0	4.46	" "

TABLE I—*Continued.*

Electrical Resistance.

No.	Bar	Composition	As Forged		After Treatment		
			Mohs. per Cm.^{3}	Rela. Res.	Mohs. per Cm.^{3}	Rela. Res.	Condition
		MISCELLANEOUS—Con.					
247	144Z	5Cr 6Mo 10Mn 10Ni 1C	63.8	5.27	78.0	6.45	Quench 1000
248	154E	2 Cr 10 Ni	44.9	3.71	45.8	3.78	" "
249	154F	5 Cr 10 Ni	55.7	4.60	59.4	4.91	" "
250	154A	7 Cr 10 nI	64.4	5.32	67.3	5.65	" "
251	154G	10 Cr 8 Ni	68.4	5.65	67.3	5.56	" "
252	154K	10 Cr 10NI	66.9	5.44	33.2	2.74	" "
253	154H	10 Cr 10 Ni 1 C	73.8	6.10	69.7	5.76	" "
254	154I	10 Cr 10 Ni 1 Si	74.2	6.13	70.9	5.86	" "
255	154B	10 Cr 10 Ni 2 Si	78.8	6.51	76.2	6.30	" "
256	146L	10 Cr 12 Ni	99.6	8.23	62.5	5.17	" "
257	137S	10 Cr 20 Ni 2 Si	93.0	7.68	91.8	7.59	" "
258	136V	20 Cr 10 Ni 1 Si 0.3 V	76.0	6.28	75.7	6.26	" "
259	126F	10 Ni 5 Mo	44.6	3.68	47.5	3.92	" "
260	146Q	3.3 Ni 7.7 Mo 2 Si	53.6	4.35	52.4	4.33	" "
261	146S	10 Ni 10 Mo 1 C	52.7	4.35	49.7	4.11	" "
262	154J	10 Ni 10 Mo 0.5 C	55.6	4.60	50.5	4.17	" "
263	146R	20 Ni 10 Mo	75.8	6.26	76.9	6.36	" "
264	155Z	20 Ni 10 Mo 1 C	79.6	6.57	78.7	6.5	" "
265	155Y	10 Ni 5 W	40.6	3.36	40.7	3.36	" "
266	155AB	10 Ni 5 W	25.4	2.10	22.4	1.85	" "
267	156A	15 Ni 5 W	45.8	3.78	44.9	3.71	" "
268	155P	1 Mn 2 Mo	22.3	1.84	20.6	1.70	" "
269	156C	1 Mn 10 Mo 0.5 C	32.4	2.68	30.4	2.52	" "
270	156N	2 Mn 5 Mo 0.6 C	35.6	2.94	44.0	3.64	" "
271	136X	10 Ni 2 Mn 2 Mo 0.5 C	64.2	5.30	63.5	5.25	" "
		2 Ni 12 Mn 6 Mo 0.8 Si	68.5	5.66	67.1	5.55	" "

Iron.

There is considerable variation in the relative conductivities of the nine iron bars tested, varying from 0.88 to 1.19, as compared with the standard 117A, which is taken as 1.0. Even the Swedish anode bar (NSW) has a higher conductivity than the standard, while some of the electrolytic forged bars are of somewhat higher relative resistance. The standard is, therefore, a good average bar.

Annealing in general causes a decrease of resistance, but the decrease is slight. Likewise, because of the fact that the average resistance of the quenched bar is no higher than the annealed, quenching appears to have little effect.

Aluminum.

But three bars containing this element were available for testing. In all cases, although the percentage of aluminum is small, there is a considerable increase of resistance. It is not possible from this small amount of data to draw any deductions in regard to the effect of varying percentages of aluminum. In the case of the one bar tested under two conditions of heat treatment, there is a decrease of resistance after annealing.

Antimony.

Because of difficulty in forging, it was possible to secure but one bar of the antimony series, with an added content of 1 percent of this element. Analysis has not been made to confirm this percentage, but it is evident that some antimony is present in the bar, because of the increase of its resistance over that of the standard, amounting to 67 percent.

Arsenic.

Referring to the six bars in this series for which analyses have given the arsenic content (121I, 121H, 113A, 113B, 113D and 113H), it will be seen that there is an increase of resistance due to the presence of arsenic. The resistance is a function of the arsenic content, and for the larger percentages of 3.86 and 3.56 in Bars 113D and 113H, it has become respectively 3.07 and 3.82 times the resistance of the electrolytic iron standard.

For Bars 92A, 113C and 113E no analyses have been made to determine the arsenic content; the increase of resistance, however, enables one to predict that there is some arsenic present, but probably not in the quantities added, particularly in Bar 113E, since its relative resistance has only increased to 1.72.

Likewise it is evident that some (but not all) of the 5 percent of arsenic remained in the transformer iron to which it was added, since comparison of Bars 172C and 172D shows an increase of electrical resistance from 18.7 to 30.2 microhms per cubic centimeter, a rise of 62 percent.

This high electrical resistance in those materials of high arsenic content is a most desirable accompaniment of the high permeabilities noted in the discussion of the magnetic tests.

Bismuth.

There is but little increase of resistance in the five bars of this group, and an irregularity of this resistance according to the added bismuth content. Since analyses have not been made, it is not possible to draw any conclusions regarding the effect of bismuth. The high conductivities of all of the samples, differing but little from the standard, points to the fact that but very little bismuth has remained in the bars tested.

Chromium.

In this group of alloys the resistance of all bars is fairly high, and in many instances it is very high, with the relative resistance exceeding five times that of electrolytic iron. Among those bars with chromium alone alloyed with iron, the relative resistance increases from 2.21 for Bar 137C (3.27 percent CR) to 4.13 for Bar 137H (16.57 percent Cr).

The highest resistances are reached in those bars having silicon present in addition to chromium; also the resistance is highest in those bars with highest silicon content. The maximum relative value of 8.50 is reached in Bar 112A, with 10 percent Cr, 5 percent Si.

The effect of vanadium is not very pronounced; there is a slight tendency towards increase of resistance with increase of vanadium. Also there is but little change in the chrome-vanadium alloys after quenching; to the contrary, the magnetic coercive force was greatly increased, as was noted in a preceding discussion of permanent magnets. This lack of alteration of the resistance is at variance with the general effect, which may be verified by comparison of the tables for electrical resistance (Table I) and for permanent magnets,[1] that the resistance changes follow the changes in coercive force and physical hardness due to annealing or quenching.

Cobalt.

In the seven bars of this group, varying in cobalt content from 0.32 percent to 6.02 percent, there is but little increase of resistance over that of the standard, and no regularity of increase or decrease due to variations of the cobalt present.

[1] These Transactions **18**, 264 (1910).

Copper.

Among the binary alloys of copper and iron there is but little change in the conductivity up to a content of copper of 7 percent, the limit of forgeability; and in general the resistance is slightly higher than for iron alone. At the other end of the series, those alloys having 90 percent or upwards of copper have resistance somewhat less than half that of iron; thus the presence of small amounts of iron to the copper decreases the conductivity very markedly.

The further addition of silicon, aluminum, tin or arsenic to the copper-iron alloys increases the resistance, and, particularly in the case of silicon, the increase varies with the increase of added element.

Annealing has resulted in a considerable betterment of the conductivity.

Lead.

But one alloy of lead and iron was tested, and analysis indicated only a small content of lead (0.061 percent). The resistance of this bar is slightly higher than the standard by 15 percent.

Manganese.

In this group are listed only binary alloys of manganese and iron, with a content of added element varying from 0.50 to 10.42 percent. It will be noted that there is a successive increase of resistance with increase of manganese, from the relatively low value of 1.33 for Bar 123H to the relatively high value of 4.57 for Bar 123J. Plotting the relative resistance as a function of the manganese percentage gives a resultant curve which is practically a straight line.

In the few cases for which tests have been made under varying heat treatment, but little change has resulted from annealing or quenching.

Molybdenum.

The alloys of this group have a higher resistance than pure iron, but the increase is not very great. Also, the conductivity is but little lowered by the greater quantities of molybdenum, as

is evidenced by the relative values of 2.30, 2.47 and 2.90 for Bars 155N (5.12 percent Mo), 155M (9.84 percent Mo) and 155 O (13.69 percent Mo).

Additions of vanadium and carbon have but little influence on the conductivity, and there is no change upon quenching, except for those bars containing carbon, where the electrical resistance increases in conformity with the increase of physical hardness.

Nickel.

A most interesting study is that of the electrical resistance of the binary series of nickel-iron alloys. The data from Table I are plotted in Fig. 1. There is a gradual increase of resistance with increase of nickel, from a relative value of 1.08 in Bar 144C (0.27 percent Ni) to 3.20 in Bar 166G (22.11 percent Ni). Here there is a sudden rise in the curve at compositions corresponding to those of the non-magnetic alloys. The maximum values are reached at 28.42 percent Ni (Bar 166C) and 35.09 percent Ni (Bar 166L). At these two points the resistances are respectively 82.0 and 81.1 microhms per cu. cm., or 6.77 and 6.70 times the resistance of the standard. The three non-magnetic bars, 154S, 166I and 166C, have the high relative resistance of 5.22, 5.41 and 6.77.

Above 35 percent of nickel the two determinations made indicate a fall of resistance with increase of nickel in a curve very similar to that on the other side of the maximum range. The relative resistances are 3.69 for 47.08 percent Ni (Bar 166 O) and 1.83 for 75.06 percent Ni (Bar 166Q). The final point of the curve at a nickel content of 100 percent is taken from a standard hand book.

Might not the maximum point in the curve of resistance be at 34 percent nickel, corresponding to the compound Fe_2Ni? Measurements of other kinds, such as dilatation, indicate a change of properties at this composition and tend to support the above view.

Vanadium appears to have but little influence on the conductivity, and there is no change due to quenching. However, in the one bar (144P) containing silicon, the resistance has reached the fairly high value of 4.50 in the forged condition, and has increased to the very high figure of 6.03 after quenching, due, no doubt, to the hardening effect of the 1 percent of carbon present.

Selenium.

The selenium content of the two bars tested is very small (0.017 percent and 0.019 percent), and no conclusions can be drawn from the data at hand. The resistance is higher, however, than the standard, the increase being 34 percent and 21 percent for the two samples.

Silicon.

The effect of additions of silicon to iron is a most marked increase of electrical resistance, an increase which is a function of the silicon content. Bars 96H (4.65 percent Si) and 109D (6.2 percent Si) have the high relative values of 5.13 and 6.18 after annealing. Since these silicon percentages are about those desired in the high permeability steels so much used in electrical machinery, we have in this alloy the additional valuable property of high electrical resistance.

Annealing of the several samples has resulted in a decrease of the resistance.

Silver.

Of the eight iron-silver alloys listed in this group, analyses of seven show so little silver present, and this amount so nearly constant in all bars, that no conclusions can be drawn from the data. The relative resistances differ but slightly from those of the standard electrolytic sample.

Tin.

In the five analyzed iron-tin alloys listed in this group, we note the increase of resistance with increase of tin. Bar 121J, with 2.06 percent Sn, has the relative value of 2.46. The highest value reached is 3.92, in Bar 110D, with an added tin content of 5 percent. Although this sample has not been analyzed, the high relative resistance indicates a high tin content; and the magnetic tests data previously discussed confirm this deduction.

In this series, therefore, we again have the desirable accompaniment of a high electrical resistance with high magnetic permeability.

Tungsten.

There is a slight increase of electrical resistance due to the addition of tungsten to iron; however, the effect is not very marked, and the relative resistance has been increased to only about 1½ times that of iron with an addition of about 24 percent of tungsten.

Also, the further addition of vanadium and carbon has but little effect. Annealing results in a slight increase of conductivity, but quenching causes very little change, except in those bars containing carbon, in which case there is a marked increase of resistance as the accompaniment of physical hardening.

Miscellaneous.

In this group appear a large number of alloys containing various combinations of chromium, manganese, molybdenum, nickel and tungsten, which promote self-hardening in steels. In a previous discussion of materials for permanent magnets, this group furnished a large part of the suitable materials.

Examination of the table shows that almost all the materials are of comparatively high resistance; and a comparison of this table of electrical resistance with Table III[2] of data for permanent magnets indicates an intimate correlation between these properties. In general those samples having physical hardness and high coercive force have high electrical resistance. Likewise, quenching has again had a varied effect, causing a decrease of resistance, or little change, or an increase; if the change has been marked, it is in general an increase of resistance. This is in confirmation of the results of the tests for permanent magnets.

As to the effect of the various combinations, the highest general resistance is attained with the nickel-chrome alloys, either alone, or with silicon, carbon or vanadium present. Nickel-molybdenum and nickel-tungsten alloys do not reach such high resistances except where the nickel content becomes so high that its presence alone results in high resistance. Also, chromium together with tungsten or molybdenum has not a very high resistance except where silicon is present; and the chrome-silicon alloys have high resistances without the added tungsten or molybdenum.

[2] These Transactions **18**, 264 (1910).

Conclusions.

Summarizing, it appears as a result of these tests that all three of the elements of arsenic, silicon and tin, which are beneficial to

TABLE II.

High Electrical Resistance.
Relative Above 5.

No.	Bar	Composition	Microhms per Cm.[3]	Relative Resistance	Condition
1	112A	10 Cr 5 Si	103.0	8.50	Forged
2	154B	10 Cr 12 Ni	99.6	8.23	"
3	146L	10 Cr 20 Ni 2 Si	93.0	7.68	"
4	126A	10 Cr 5 W 2 Si	92.2	7.61	"
5	141E	15 Cr 2 Si	92.6	7.55	"
6	126B	7.23 Cr 9 W 3 Si	89.5	7.39	"
7	122E	10 Cr 3 Si	87.5	7.23	"
8	137L	10 Cr 2 Si 2 Ti	86.0	7.11	Quenched
9	133N	13 Cr 1.8 Si	85.6	7.08	"
10	141C	10.3 Cr 1 V 2 Si 0.3 C	84.9	7.01	"
11	156P	10Cr 2W 2Mo 2Si 0.3V	83.8	6.93	"
12	141G	21.4 Cr 3 Mn 1 Si 1 V	82.0	6.77	"
13	166G	28.42 Ni	82.0	6.77	"
14	166L	35.09 Ni	81.1	6.70	"
15	146R	20 Ni 10 Mo 1 C	79.6	6.57	Forged
16	154I	10 Cr 10 Ni 2 Si	78.8	6.51	"
17	126D	2 Si 10 V	78.9	6.51	"
18	140Q	5Cr 6Mo 10Mn 10Ni 1C	78.0	6.45	Quenched
19	155K	10 Cr 2 Si 0.3 V 0.6 C	77.4	6.40	"
20	109O	6.2 Si	77.2	6.37	Forged
21	154J	20 Ni 10 Mo	76.9	6.36	Quenched
22	136H	17 Cr 1½ Si	76.0	6.28	"
23	137S	20 Cr 10 Ni 1 Si 0.3 V	76.0	6.28	Forged
24	111S	10 Cr 2 Si	75.0	6.20	"
25	163O	10 Cr 1 V	74.8	6.18	Quenched
26	154H	10 Cr 10 Ni 1 Si	74.2	6.13	Forged
27	154K	10 Cr 10 Ni 1 C	73.8	6.10	"
28	156K	10 Cr 2 W 2 Si	72.8	6.02	Quenched
29	146P	10 Ni 2 Si 1 C	70.3	5.81	Forged
30	136X	2 Ni 12 Mn 6 Mo 0.8 Si	68.5	5.66	"
31	154F	7 Cr 10 Ni	67.3	5.65	Quenched
32	154A	10 Cr 8 Ni	68.4	5.65	Forged
33	136L	20 Cr 3 Mn 1 Si	67.1	5.55	Quenched
34	154G	10 Cr 10 Ni	66.9	5.44	Forged
35	166I	26.40 Ni	65.5	5.41	Quenched
36	133F	6 Cr 1.68 Si	64.4	5.32	"
37	154S	25.20 Ni	64.2	5.30	Forged
38	154S	25.20 Ni	63.2	5.22	Quenched
39	96H	4.65 Si	62.2	5.13	"
40	156M	10 Cr 2 W 2 Mo 0.6 C	61.6	5.09	Forged
41	133M	8 Cr 1 Si	61.2	5.06	Quenched
42	155V	10 Cr 10 W	60.7	5.02	Forged
43	156I	10 Cr 2 W 0.3 V 0.6 C	60.4	5.00	Quenched

the magnetic properties of iron, have the desirable accompaniment of high electrical resistance.

Among the materials adapted for high resistances, various combinations of iron with nickel, chromium and silicon are most suitable.

The change of resistance due to heat treatment in general follows the changes of physical hardness due to the treatment. Annealing usually results in a decrease of resistance. Quenching is uncertain in its effect; usually there is an increase of resistance with physical hardening of the bar.

High Resistance Materials.

There are grouped in Table II, in the order of their relative resistances, all those iron alloys tested which showed a relative value as compared with the electrolytic iron *of five or upwards.* The highest resistance recorded is 8.50, reached by Bar 112A, with 10 percent Cr, 5 percent Si. Examination of the table shows 43 bars listed, all of which contain chromium, nickel or silicon, either alone or in various combinations.

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DISCUSSION.

MR. C. A. HANSEN: The following specific resistance on the high nickel end of the nickel-iron series may be of interest as supplementing the results given in this paper. These measurements were made on drawn wire by the Standardizing Laboratory of the General Electric Company:

	Percent Fe	Percent C	Percent Si	Microhms per Cm.^{3} at 20° C	(20°–90° C)
Nickel.....	...	.071	.170	16.3	.00259
"	...	.110	.640	19.7	.00222
" iron.....	3.70	.120	.350	20.0	.00265
" "	7.14	.110	.260	19.6	.00242
" "	10.34	.110	.094	21.4	.00273
" "	18.75	.190	.014	24.1	.00325

DR. E. F. NORTHRUP: Were these resistance measurements made at room temperatures?

DR. O. P. WATTS: All were made at room temperatures of the laboratory, which would be about 23° to 24° C.

MR. CARL HERING: Do you know how he made the arsenic alloys, as arsenic volatilizes so readily?

DR. WATTS: The material was weighed out into these crucibles, put inside the graphite crucible, then a graphite cover placed over it, and the crucible placed in a granular resistor furnace, in which it was allowed to cool, after the metal was melted.

MR. HANSEN: I think I have seen that arsenic series before, where he has put 10 percent arsenic in the alloy and given an analysis of 3½ percent. In this series he seems not to have given any analysis, but gave the added percentage. Another thing we are particularly interested in is that all those bars were forged.

DR. WATTS: All bars were forged.

PRESIDENT WHITNEY: That means you can forge a bar of 10 percent chromium and 5 percent silicon?

DR. WATTS: Yes, all these bars were forged. I have seen a hundred other bars smashed before my own eyes.

MR. HANSEN: I think the list of the hundred alloys that were smashed, if we could have a little amplifying detail, might be as valuable as the list of materials forged would be to the people who are interested in making electrical resistance alloys.

DR. WATTS: Those must be placed in the series of which the President spoke this morning, of unsuccessful experiments. We have not published them as yet.

MR. JAMES ASTON (*Communicated*): Referring to the discussion of the results given in the paper, it must be confessed that they are open to criticism from the standpoint of exact physical measurement, in that no endeavor was made to correct for slight temperature changes; also that the method does not approach the accuracy of Wheatstone bridge or potentiometer measurements. No apology is necessary, however, since the data was obtained and put forth not as being the last word on the subject, but to give the general effect of the various alloying elements on the conductivity of the pure iron base.

We regret that two errors crept into the paper as presented, one of which has been corrected in this final publication. In the description of the method employed, there was the following paragraph: "The apparatus used consisted of two knife edges, 7.854 cm. apart, upon which the test bar was laid, and pressed into firm contact by means of spring clips; these also served as conductors to lead the current into the bar." Interpreting this to mean that the knife edges served as the current conductors, and in view of the fact that large currents (50 amperes) were often used, it is evident that there would be marked errors when these same knife edges were also the contacts for the millivoltmeter connections. However, this was not the case, since the spring clips which pressed the bar firmly into contact with the knife edges were the current conductors, and the knife edges which were between or inside the spring clips carried only the small current taken by the millivoltmeter. This ambiguity has been eliminated by making the paragraph read as follows: "and pressed into firm contact by means of spring clips; the latter were outside of the knife edges, and served also as conductors to lead the current into the bar."

The uncorrected error is in plotting the curve for the nickel series, in that the point at 81 microhms per cubic centimeter, applying in the tables to the alloy with 35 percent Ni, was plotted as 30.5 Ni.

In regard to the iron-arsenic alloys, they were made by a simple melting of the two materials in a magnesia crucible, as explained by Dr. Watts. Although arsenic volatilizes long before the melting point of iron is reached, the analyses of the alloys agreed very closely with the added proportions up to 4 percent As. This seems to be the saturation point under the conditions of melting, and although added amounts up to 10 percent were tried, the highest percentage on analysis was 4.14 percent.

All bars for which results are given were forged and turned into shape. In many cases added percentages only are given as the composition of the bars; but, as mentioned in the paper, the plotted data is all from analyzed bars, and most of the others are alloys of iron with such elements as are taken up in practically the added proportions.

A paper presented at the Twentieth General Meeting of the American Electrochemical Society, in Toronto, Canada, September 22, 1911, President W. R. Whitney in the Chair.

THERMAL CONDUCTION AND CONVECTION IN GASES AT EXTREMELY HIGH TEMPERATURES.

By Irving Langmuir.

In 1904, Prof. W. Nernst, then at the University of Göttingen, suggested as a thesis subject the study of the formation of nitric oxide from air in the neighborhood of a glowing Nernst filament. It was thought that the filament would probably act catalytically on the reaction between oxygen and nitrogen, and that in a closed vessel, after a stationary condition had been reached, the amount of nitric oxide would be that corresponding to the equilibrium

$$N_2 + O_2 = 2NO$$

at the temperature of the filament.

Experiments showed, however, that this was far from being the case. The rate of formation of nitric oxide was extremely slow, and the percentages obtained were only a small fraction of those corresponding to equilibrium. After carrying out a considerable number of experiments, work along this line was discontinued and reactions in which the filament would more surely act catalytically were studied. The following reactions

$$2H_2O = 2H_2 + O_2$$
$$2CO_2 = 2CO + O_2$$

when brought about by glowing platinum wires, were found to be of this type, and it was possible in this way to determine the equilibrium constants of these reactions with great accuracy.[1]

The earlier experiments with the Nernst filaments, however, had given some very peculiar results and had led to a more extended theoretical investigation into the nature of reactions around

[1] J. Am. Chem. Soc. 28, 1357 (1906).

glowing filaments. The results were published as a dissertation,[2] but as some of the theoretical conclusions needed more experimental evidence to support them, the final publication of the results has been delayed until the present time.

Experiments with Nernst Filaments in Air.

Clement[3] showed that nitric oxide without even a trace of ozone is formed when a Nernst filament glows in air. The object of the experiments about to be described was to find how nearly the concentration of nitric oxide produced in this way agreed with that found by other methods at temperatures corresponding to that of the filament.

A few preliminary experiments showed that the nitric oxide was formed extremely slowly; the highest rate of formation observed was only 1.4 c.c. of nitric oxide per hour. The concentration gradually rose until after many hours it became practically constant. It was found that the results of the experiments at any temperature could be expressed by two quantities: 1st, the percentage of nitric oxide in the gas when a stationary condition (or "pseudo-equilibrium") was reached; 2nd, the rate at which this equilibrium was reached. The reaction proceeded according to the laws of a reaction of the second order in agreement with Nernst.[4]

Two methods were used in studying this pseudo-equilibrium and the velocity with which it was approached.

First Method. A very slow stream (0.006 to 0.1 c.c. per second) of air dried over phosphorus pentoxide was passed through a small bulb containing the glowing filament. The nitric oxide was absorbed in concentrated sulfuric acid and then evolved and measured by shaking the sulfuric acid over mercury in a gas burette.[5] If u be the rate of flow of the air current (c.c. per second), x the fraction of nitric oxide in the air after passing the filament, a the limiting value of x when $u = o$, and H the velocity of the reaction around the filament, then the following formula enables a and H to be calculated.

$$H\ (a^2 - x^2) = ux$$

[2] Ueber partielle Wiedervereinigung dissocierter Gase im Verlauf einer Abkühlung. Inaugural Dissertation, Göttingen, 1906.
[3] Ann. Phys. **14**, 334 (1904).
[4] Zeit. anorg. Chem. **49**, 224 (1906).
[5] Lunge's method.

This equation, as well as those given below, was deduced theoretically. The details of the calculation will not be gone into here; let it suffice to say that H is related to the ordinary velocity coefficient k as follows:

$$H = T_z \int \frac{k}{T} de$$

where k is the velocity coefficient in the element of volume de

T is the temperature (absolute) in the element of volume

T_z is room temperature (absolute).

Second Method. The glower was placed in a vessel of about one liter capacity containing dry air. After glowing the filament for a period of time ranging from 4 to 43 hours, it was allowed to cool and the gas was then drawn out of the vessel through an absorption tube containing sulfuric acid. The nitric oxide was determined as before. The values of a and H were then calculated from the following equation

$$t = \frac{V}{2Ha} \ln \frac{a + x}{a - x}$$

where t = time during which the filament is heated

V = volume of air in the vessel

H, a and x have the same significance as before.

The temperature of the filament was determined by measurement of the luminosity in candle-power per square mm. of surface, and the application of Rasch's equation (see below).

The numerical results are briefly summarized in the following table:

Experiment No.	32-34	40	22-27	41	35-36	42
Filament No.....	8	9	5	9	8	9
T_0 (abs.)	2320	2358	2373	2380	2395	2426
1000 a.....	2.36	3.64	4.35	4.49	6.51	7.42
1000 x_0.....	12.8	13.8	14.2	14.4	14.9	15.7
H per cm. of filament	7.2	—	6.2	—	3.4	—

1,000 a is the amount of nitric oxide (in c.c. per liter) present when the pseudo-equilibrium has been reached. The 1,000 x_0 is the amount of nitric oxide[6] corresponding to true equilibrium at the temperature T_0 of the filament.

[6] Nernst, l. c.

These results are remarkable in three respects:

I. The values of a are very much lower than the corresponding values of x_0. It is to be expected that they would be somewhat lower, but the amount of the difference is surprising. For example, in the experiments at 2320° K.,[7] only 0.2364 percent of nitric oxide is formed. This corresponds to the true equilibrium at about 1700° K., so that in all those parts of the space where the temperature is above 1700° K., nitric oxide must be forming, and this must all be decomposed again in those parts where the temperature is below 1700° K. As the velocity of the reaction drops off very rapidly with falling temperature, it follows that the volume of gas which is nearly at the temperature of the filament is *very* small compared to that at a temperature slightly below 1700° K.

II. The values of a approach those of x_0 with extraordinary rapidity as the temperature rises. Just the opposite would be expected. That is, it would seem most natural that the higher the temperature of the filament, the greater would be the difference between the observed amount of nitric oxide and that corresponding to the true equilibrium.

III. The velocity coefficient H decreases with increasing filament temperature. The quotient $H \div V$ is the average reaction velocity in the whole vessel containing the filament. Since the true velocity coefficient increases rapidly with the temperature (Nernst, l. c.), the only way in which H can decrease is for the volume of the hot gases around the filament to decrease as the temperature of the latter rises.

This naturally suggests that the temperature gradient around the filament increases rapidly as the temperature of the filament rises, and this would lead to a much increased loss of heat by conduction through the gas. It is possible that an increase in the heat conductivity of the air would lead to very much increased convection and thus cause an actual lowering of the temperature of the outer layers of gas. This explanation of the third peculiarity of the reaction around the Nernst filament helps greatly in understanding the first and second. All three can be explained in the following manner:

[7] In this paper the letter K (Kelvin) will be used to denote absolute temperatures in accordance with the recommendations of the Association International du Froid. See Chem. Ztg., **35**, 3 (1911).

At $T_0 = 2320$, the volume of gas slightly below 1700° is so much larger than that near the temperature of the filament that, in spite of the much lower velocity coefficient, the reaction takes place principally in the portions of the gas with a temperature of about 1700°.

At $T_0 = 2420$, the heat conductivity of the gas has increased so much that more heat is carried from the filament and thus more vigorous convection currents are set up. The volume of the very hot gas probably increases, whereas the volume of the gas with a temperature around 1700° decreases. The decomposition of the nitric oxide in the outer layers has therefore decreased, while the rate of formation near the filament has increased. This causes the extraordinary increase in a. On the other hand, the rate of formation of the nitric oxide in pure air is less than before because of the marked decrease in the volume of the reacting gas. In the dissertation referred to above, some simple assumptions were made as to the distribution of temperature around the filament, and equations were then deduced which gave a very satisfactory quantitative method of calculating H from the ratio between a and x_0.

The above hypothesis of the rapid increase in conductivity or convection at high temperatures received a further strong support by a study of the characteristics of the Nernst filaments.

Volts, amperes and candle-power measurements were carefully made by Mr. Schlüter, Prof. Nernst's assistant, on about a dozen glowers. The temperatures were calculated by Rasch's equation, assuming that the emissivity for visible radiation is the same as that of a black body. This is not strictly accurate, but even if it should emit only 75 percent as much as a black body, the error in the temperature determination would be only 52°.

The watts supplied to the glowers in every case varied with a power of the absolute temperature lying between 4.4 and 5.1.

According to Nutting,[8] the total *radiation* from a Nernst glower when at about normal brilliancy varies with the 4.3 power of the absolute temperature. Even if the filament radiated as much as a black body, the radiation would amount to only about two-thirds of the whole energy, the rest being lost thru the air. In all probability the radiation is somewhat less than one-half of the

[8] Bull. Bur. Standards 4, 550 (1908).

total energy. Since the total energy varies with the 4.4 to 5.1 power, and the radiation varies as the 4.3 power, the energy conducted away by the air and through the leads must vary with at least the 4.5 to 5.9 power of the absolute temperature.

This seems a remarkably high value, but a rough calculation shows it to be of about the right order of magnitude to account for the peculiarities observed in the formation of nitric oxide around a Nernst glower.

It is usually considered that one of the most noteworthy characteristics of radiation is the fact that it increases so rapidly with the temperature. The common experience is that conduction and convection are the principal causes of temperature equalization at ordinary temperatures, but as the temperature increases, radiation very soon predominates. By Newton's law of cooling, the loss of heat is proportional to the difference of temperature between an object and the surroundings. It has long been known that this law is only roughly approximate. Even as early as 1817, Dulong and Petit proposed the empirical law that the loss of heat by conduction and convection is proportional to the 1.233 power of the difference of temperature between the hot object and its surroundings.

The literature on the subject of the convection of heat at high temperatures is very meager, most observers having been content to carry out their experiments at temperatures not exceeding 300° or 400°. Alex. Russell[9] derives equations that agree well with experiment at moderately high temperatures.. In his article will be found references to the most important literature on the subject. Hartmann[10] made experiments with rods prepared from pressed platinum black, which he heated up to 1900° K. He finds that the loss of heat by conduction is well expressed by a formula of the form $W = k(1 + bt)t$. At very high temperatures this would give the loss by conduction and convection as varying with the square of the temperature. A fairly careful search through the literature has failed to show any cases where experiments have been carried out at a higher temperature than this or where the heat loss has been found to vary more rapidly than with the square of the temperature.

[9] Phil. Mag. **20**, 591 (1910).
[10] Phys. Zeit. **5**, 579 (1904).

An elementary consideration of the processes that are ordinarily thought to constitute convection and conduction fails to give any reason why the loss of energy by these means should ever increase more rapidly than with the square of the temperature. The driving force causing convection is the difference in weight of two columns of gas at different temperatures. Now the weight of the gas is inversely proportional to its temperature and directly proportional to the pressure, so the driving force is proportional to

$$p\left(\frac{1}{T}-\frac{1}{T_o}\right) \text{ or } p\left(\frac{T_o - T}{T_o\, T}\right)$$

The resistance to be overcome by this force is the force of friction. The coefficient of internal friction increases approximately with the ⅔ power of the temperature.[11] The amount of heat carried by convection is roughly proportional to the driving force and inversely proportional to the internal friction, and both these factors thus tend to cause a marked decrease in convection at high temperatures, that is, for a given difference in temperature between the hot body and its surroundings. This conclusion has been verified by some experiments recently made by the author which showed very clearly that, at the temperature of liquid air, convection is much more active than at ordinary temperature. For example, it was found that the pressure has to be lowered much more at liquid air temperatures than it does at higher temperatures before convection becomes small compared to conduction.

According to the kinetic theory of gases, the heat conductivity would increase with about the 0.85 power of the temperature.

If convection is the most important factor in the loss of heat from the filament, it would seem that above a certain temperature the heat loss would increase less rapidly than proportional to the temperature. On the other hand, if, at the highest temperatures, conduction should predominate, the heat loss should still not increase more rapidly than with the 1.85 power of the temperature.

These considerations lend particular interest to the conclusions drawn from the experiments on the formation of nitric

[11] See Meyer's Kinetic Theory of Gases.

oxide around the Nernst glower. If it should be a fact that the heat loss by convection and conduction from a Nernst glower at 2400° varies with nearly the fifth power of the temperature, it would seem to prove that some new and previously unsuspected phenomenon was taking place. A dissociation of the gas molecules into atoms or an absorption of energy by the emission of ions from the hot surface suggest themselves as possible causes. Either cause, however, would be of sufficient interest to warrant further investigation.

The Nernst glower is not suitable to use for these experiments, for the range of temperature over which it can be operated is too limited. No more suitable material could be found than the ductile tungsten wire made in this laboratory. In an atmosphere of hydrogen this wire can be heated to temperatures above 3000° C. for considerable periods of time. Furthermore, because of its very high temperature coefficient of electrical resistance, its temperature can be measured with considerable accuracy.

Experiments with Tungsten Wire in Hydrogen.

The tungsten wire was suspended in the axis of a vertically placed glass tube of 4 cm. internal diameter, down through which a stream of very pure electrolytic hydrogen was made to flow. The hydrogen was purified by passing it through two furnaces containing copper gauze, then dried over sulphuric acid and phosphorus pentoxide, then passed through a tube filled with paladium asbestos heated to 300° C., and finally dried again over phosphorus pentoxide. The wire was held at the upper end by a clamp, and the lower end was fastened to a heavy piece of platinum wire, which dipped into a small cup containing mercury in order to furnish electrical connection with a copper wire entering the lower end of the tube. The lengths of tungsten wire used ranged from 5 to 50 cm., and wires of four different diameters, 0.229, 0.069, 0.045, 0.028 mm., were studied.

A good many experiments were also made with tungsten wires placed horizontally, bent into loops and placed in vessels of various shapes and sizes.

Two methods were used in determining the temperatures

of the wire. First, where the temperature was sufficiently high a direct measurement of the horizontal candle-power of the wire was made by a portable Weber photometer.[12] The temperature was then calculated by means of the following formula:

$$T = \frac{11230}{4.933 - com.\ log.\ \left(\frac{10}{3}\,\frac{c}{ld}\right)}$$

where T = absolute temperature
c = horizontal candle-power (international units)
l = length of the wire in cm.
d = diameter of the wire in mils.

This formula is obtained from algebraic transformation from Rasch's equation,[13] slightly changing the constants to agree with more recent work, and making the following assumptions: That the glass absorbs 3 percent of the light; that tungsten radiates 51 percent as much light as a black body at the same temperature. This last assumption is based on some measurements made, March, 1910, by the Bureau of Standards on the reflectivity of tungsten,[13a] and is in full agreement with the published results of von Wartenberg.[14] These measurements of the reflectivity were made at ordinary temperatures, but there has been much recent work to show that the reflectivity at high temperatures is the same as at ordinary temperatures. This is especially well proven by the papers of Henning,[15] Rubens[16] and von Wartenberg.[17]

The second method consisted in the determination of the resistance (per cm. of length) of the hot wire from volt and ampere readings, and in the comparison of this with the resistance (per cm.) of wire from the same spool run at an efficiency of one watt per candle-power in an incandescent lamp. From data available in this laboratory, which will probably soon be

[12] Schmidt and Haensch.
[13] Ann. phys. **14**, 193 (1904).
[13a] Bull. Bur. Standards **7**, 202 (1911).
[14] Ber. physik. Ges. (1910), 105-120.
[15] Zeitsch. f. Instrumentenkunde, **30**, 1195 (1910).
[16] Ber. physik. Ges., **12**, 172 (1910).
[17] Ber. physik. Ges., **12**, 121 (1910).

published, it was thus possible to calculate the temperature of the wire even at temperatures so low that candle-power measurements could not be made. The two methods gave results agreeing within about 50° at temperatures as high as 3000°.

Summary of the Results.

1. The energy required to heat the wire to any given temperature is very nearly independent of the size of the vessel containing the hydrogen. As a rule the energy necessary to give a certain resistance per cm. was about 10 percent less in a large brass tube (9 cm. diameter) than it was in the glass tube (4 cm. diameter).

2. The position of the wire in the vessel makes considerable difference at low temperatures, but at high temperatures (above 2400° K.) it has very little effect. Whether the wire is vertical or horizontal or even bent into the shape of a loop makes comparatively little difference at high temperatures.

3. Even at the highest temperatures no appreciable fraction of the whole current is carried by the gas in the neighborhood of the wire when the latter is straight. If the hydrogen close to the wire should become sufficiently ionized to conduct a perceptible part of the current, it would cause a marked decrease in the resistance and thus cause a very large divergence in the temperature calculated by the two different methods. In the case of wires bent into the form of loops (with ends about 1 cm. apart), a very distinct decrease of resistance was observed when the voltage was greater than 200 volts and the temperature was 2900° K. or more. Under these conditions, but at a somewhat higher temperature or voltage an arc always formed and destroyed the wire.

4. The relation between energy consumption and temperature is given in the following table. These results are taken from smoothed curves obtained from experiments in which the wire was suspended in the axis of a vertically placed glass tube of 4 cm. diameter filled with hydrogen. The rate of flow of hydrogen within ordinary limits had no effect on the energy consumption.

Temperature absolute	Total Energy Lost by Wire, (Watts per cm. of length.)			
	Wire A Diam. .028mm.	Wire B Diam. .045	Wire C Diam. .069	Wire D Diam. .229
1000 °K	. . .	2.1	. . .	. . .
1200	. . .	3.0	3.3	5.1
1400	. . .	4.0	4.4	6.9
1600	. . .	5.3	5.8	9.4
1800	. . .	7.1	8.0	12.9
2000	8.8	9.4	10.7	17.8
2200	11.2	12.2	14.1	24.8
2400	14.5	16.0	18.9	35.0
2600	20.0	22.2	26.6	52.7
2800	28.6	32.1	39.2	. . .
3000	42.4	48.0	59.5	. . .
3200	67.3	77.3	97.5	. . .
3400	. . .	136.4	175.	. . .

According to A. Oderbeck,[18] the energy loss from small wires is independent of the diameter of the wire. This result can also be deduced from the formulas given by Peclet.[19] Hartmann,[20] also, finds the same to be true. From the above table it is seen that the higher the temperature, the more the heat loss depends on the diameter of the wire. At low temperatures (1200° K.) the effect of diameter is very slight. On the other hand, at 2400° K. the heat loss appears to be a linear function of the diameter, it being possible to calculate the energy from the equation

$$w = w_0 \, (0.71 + 6.5 \, d)$$

where w_0 = watts per cm. for wire B
d = diameter of the wire in mm.

5. The loss of energy is seen to increase very rapidly with increasing temperature. The rate at which it is increasing may be calculated by the formula

$$n = \frac{d \, (log. \, w)}{d \, (log. \, T)}$$

[18] Ann. phys. 56, 397 (1895).
[19] Traite de Chaleur 3ieme Edition, Paris 1860, page 373.
[20] Loc. cit.

The following table gives the values of n calculated in this way from the data on wire B:

Temperature absolute	Exponent n.
1000°	1.86
1500	2.08
2000	2.71
2500	4.03
3000	6.90
3400	10.1

That is, up to about 1300° K. the energy increases proportional to a power of the temperature between the first and the second, but above that it gradually increases more rapidly until at 3400° K. it is actually increasing with more than the tenth power of the temperature.

In a vacuum the energy loss is proportional to the 4.8 power of the temperature, and this exponent is constant over a very wide range. So, for a wire of about 0.045 mm. diameter, heated in hydrogen, the ratio of radiated to total energy reaches a maximum at about 2600° K. At this temperature, however, only about 9 percent of the total energy is lost by radiation, while 91 percent is carried away by conduction through the hydrogen. At 1000° K. the proportion of radiated energy is about 0.7 percent., and at 3400° it is 3.4 percent of the total energy.

These results with hydrogen strongly confirm the conclusions drawn from the experiments with the Nernst glower. The most reasonable explanation of the very large heat conductivity of these gases at these extremely high temperatures seems to be that the molecules dissociate into atoms close to the filament, thereby absorbing a large amount of heat. The atoms then diffuse away from the wire, and there, because of the lower temperature, recombine and give up the heat that was absorbed from the wire. That the dissociation of a gas causes an abnormally high heat conductivity was shown by Magnanini and Malagnini[21] in their study of the heat conductivity of nitrogen peroxide. They found that the partly dissociated gas had a conductivity three times as great as that which was completely dissociated.

Experiments are now in progress to determine the energy loss

[21] Nuovo Cim. 6, 352 (1897).

of a tungsten wire heated in monatomic gases. Whether or not the above explanation is correct will undoubtedly be shown by these experiments.

Melting Point of Tungsten.

In connection with these experiments it should be noted that the candle-power measurements indicate that the melting point of tungsten is at least 3450° K. Pirani[22] had found 3570° by a very unsatisfactory method of extrapolation, but the best value up to that time, and the one that even now seems to be generally accepted, is that of von Wartenberg, who gave 3170° K.

The author wishes to express his appreciation of the services of Dr. A. H. Barnes, who carried out much of the experimental work with tungsten wires in hydrogen.

Research Laboratory,
General Electric Company,
Schenectady, N. Y.,
August 4, 1911.

DISCUSSION.

Dr. E. F. Northrup: Do these experiments throw any light on whether or not Stefan's law, that the total radiation is proportional to the fourth power of the absolute temperature, holds true at these high temperatures?

Mr. I. Langmuir: In the case of tungsten, tungsten not being a black body, Stefan's law does not hold with that exponent. We find that the radiation, instead of increasing with the fourth power, increases with the 4.8 powers of the absolute temperature. Tungsten, as it approaches higher and higher temperatures, approaches more and more nearly to a black body condition, not in any one particular wave-length, but as the point of maxi-

[22] Ber. physik. Ges. (1910), 310.

mum energy shifts to shorter and shorter wave-lengths, it shifts into a region where the emissivity of the metal is higher. The emissivity of tungsten for infra-red rays is low. At the lower temperature it emits, probably only 20 percent as much as a black body would at the same temperature, whereas, with visible light it radiates about 50 percent as much. Consequently, as you raise the temperature, the radiation gradually shifts over from that part of the spectrum where its emissivity is around 0.2 up to a part of the spectrum where its emissivity is 0.5. That causes the energy to increase more rapidly than that of a black body.

Mr. M. G. Lloyd: Were measurements in hydrogen made at any other than atmospheric pressure? If not, I should think that might shed some light upon the relations involved.

Mr. Langmuir: Some measurements at other pressures are in progress at present. We are fitted up to do it very easily at lower pressures. I know that at a pressure down to 10 centimeters mercury or thereabouts the heat lost by the filament is not affected very much by the pressure.

I might just point out that some of these quantities of energy seem extremely high. We are dealing with current densities that are very different from what we ordinarily think of. One piece of wire a little over 0.001 inch in diameter, or 1.9 mils in diameter, carries about six amperes before it burns out, which means about 175 watts of energy per centimeter of length, or 11.5 kilowatts per sq. cm. of surface, about 30 times as much as the same wire would carry in a good vacuum, at the same temperature. This is as much energy as would be dissipated by a black body in a vacuum at a temperature of 6500° C.

One other point is the advantages of carrying out melting-point determinations of tungsten in an atmosphere of hydrogen. There are three very distinct advantages. In the first place, the fact that the energy goes up with nearly the tenth power of the absolute temperature, instead of the 4th or 5th power of the absolute temperature, means just this: You can get a great deal closer to the melting point without having a wire burnt out. In a vacuum, when you heat a wire close to the melting point, if one spot gets a little thin from some cause (either a little thin

to begin with, or by evaporation of the metal), that would cause the temperature to rise a little at that point. In hydrogen the energy loss increasing with the tenth power of the absolute temperature, the temperature will not rise above half as much above that of the rest of the wire as in a vacuum itself. This is one advantage.

The second is that the tube in which the experiments are made does not blacken up, and therefore does not interfere with candle-power measurements. In a vacuum, if you get the filament right close to the melting point of the bulb, it will blacken while you take the measurements. You cannot take measurements at the melting point of tungsten and get a candle-power determination worth anything. Thirty or forty percent of the light is absorbed by the deposit on the bulb.

The third is that the energy loss does not vary much with the diameter of the wire. If a wire does get thin in one place, it still gives out just as much energy as though it were larger, whereas, in a vacuum, as soon as the diameter of the wire at any point goes down to one-half its original value, it loses only half of the energy at that point, and therefore gets a great deal hotter; in other words, the temperature rises very much more rapidly in a vacuum than in hydrogen.

MR. CARL HERING (*Communicated*): Mr. Langmuir's interesting paper involves some subjects which, in my opinion, have not been given the importance they seem to deserve. I refer more particularly to the general subject of the transmission of energy from a highly heated body to its surroundings, which is of such great importance in the economical operation of electric furnaces.

I was pleased to see that, unlike many other writers, he refers to this flux as *energy,* in distinction from *heat,* although in the title and body of the paper he does use the words "thermal" and "heat loss," which seem less consistent. The point I wish to emphasize is that, in my opinion, it leads to confusion and errors, and may even sometimes be quite wrong, to assume, as is so often done, that these so-called "losses" of energy from a highly heated body are necessarily always in the form of heat, and must therefore be accounted for by either radiation (of heat as such), conduction (true) and convection (of heat as such). Nor should

they be called "losses," in general discussions of the laws; in an arc radiation furnace, for instance, these so-called "losses" constitute the useful energy; in an incandescent lamp these "losses" form in part the desired light. Why not call it, broadly, a transmission of energy, and then add how it has been transmitted.

Radiant heat as such is an indefinite form of energy, more indefinite even than light, the latter being at least defined, in its wave-lengths, by the particular retina which the human eye has, that is, by the "visible" spectrum. When such wave-lengths of ether vibrations are very long, such as those used in wireless telegraphy, they can certainly not be classed as heat waves; and, on the other hand, when they are as short as in and beyond the ultra-violet part of the spectrum, their heat value again becomes very slight; nor should heat radiations as such be confounded with other radiations, like light, which are converted into heat after having been radiated. Hence heat radiation at best is an indefinite and therefore unsatisfactory term to use in a quantitative sense.

It has often suggested itself to me, and it does not seem unreasonable, that at such very high temperatures the energy which leaves a hot body may be in part, and perhaps even largely, energy of very short wave-lengths, like that in and beyond the ultra-violet part of the spectrum. If so, does it necessarily follow that the transmission of such waves, or of such a form of energy, must follow the laws of heat, that is, those of thermal radiation, conduction and convection? May not the passage or transmission of such energy follow other laws? If so, does it not create confusion to try to explain it by thermal laws or to reduce it to those laws?

A true resistance to heat radiation seems hard to conceive, and is apparently not used in text books, yet a resistance to light radiation is implied in the terms transparent, translucent and opaque; is it known whether it exists for the still shorter waves? Roentgen rays are known to meet with less "resistance" in passing through the human body than light rays do; ultra-violet rays seem to cast no shadow of the human body, hence meet no resistance in it; some white bodies reflect them completely (hence have a low virtual resistance). In the passage of radiant energy

through a body, can its subsequent transformation into local heat, generally called absorption, be properly referred to as being due to a resistance? Or can a body be said to offer a high resistance when it reflects such energy? Sometimes such absorption produces chemical changes instead of heat.

If such a highly heated tungsten wire as that referred to in the paper gave off a relatively large quantity of energy of very short wave lengths, which seem likely, does it not, in view of the above, create confusion to try to account for it all by radia-

short wave lengths, which seems likely, does it not, in view of these is calculated by the difference between the total and the other two? Mr. Langmuir has himself suggested another possible means of transmission, namely, by a storage of energy in the medium near the wire in the form of a dissociation of molecules into atoms, and a subsequent setting free of this energy as heat after these atoms have traveled bodily to a cooler region. This energy might therefore be said to be transmitted through space in the form of chemical bonds which combine atoms, just as some of the energy of Niagara Falls is transmitted through space by the railroads, in the form of calcium carbide (hence *not* as heat), to be set free as light and heat somewhere else. Such a transmission seems to be more akin to convection than to conduction, tho quantitatively the laws of convection would not seem to apply correctly; Mr. Langmuir, however, seems to call this kind of transmission "conductivity," which does not seem to be a consistent use of that term.

This subject may have an important bearing on arc furnaces, particularly on those depending upon the radiations from the arc. Is it not likely that an arc sends out many short-wave chemical rays, or perhaps chemically active ions, besides mere heat and light, and may it not be that this chemically active energy plays an important part in sweating the roof of an arc furnace by chemical action rather than by temperature only?

It seems to me that the general subject of the transmission of energy from hot bodies to the mediums surrounding them is of sufficient practical importance to warrant more careful and more systematic study than has been given it, and less looseness in our terms.

Mr. Langmuir states that "the rate of flow of hydrogen within ordinary limits had no effect on the energy consumption." And yet he says later that, of the total energy, "91 percent is carried away by conduction through the hydrogen." Heat conduction, as I understand it, refers to a sort of flow of heat energy from one part to another, just as water flows through a sponge or porous material; hence, if that material itself also moves away, such a movement ought to increase this flow, hence increase the apparent conductivity. The first statement seems to say it does not, yet the second says that almost the entire energy is transmitted by conduction, very little by radiation, and none by convection. His use of the term conduction seems unfortunate.

A paper presented in abstract at the Twentieth General Meeting of the American Electrochemical Society, in Toronto, Canada, September 22, 1911, President W. R. Whitney in the Chair.

ON THE MEASUREMENT OF VERY SMALL GAS PRESSURES.

By C. F. Hale.

The extension of studies of the phenomena in rarified gases to the region of very low pressures has made increasingly important the development of manometers whose indications of pressure are not dependent upon the use of mercury. Thus, with the modern rotary pump it is a relatively simple matter to evacuate glass vessels to a pressure of 0.01 mm. mercury, but if a McLeod manometer is employed to measure the residual pressure, the mercury in it contributes about 10 percent to the total pressure, and, furthermore, the manometer does not indicate the pressure of the vapor which it contributes; its reading is the partial pressure due to other gases, and not the total pressure. In some experimental work, too, it is imperative that mercury vapor be excluded from the apparatus.

During the last few years a number of non-mercury manometers have been described in the literature. Ladenburg and Lehmann[1] have applied the Bourdon Spiral to the measurement of small changes in pressure, and later it was employed by Johnson[2] in the investigation of dissociation phenomena with ammonium chloride. This manometer consists in a flat tapered glass tube, bent into the form of a spiral. The wider end of the tube is directly connected to the system, the pressure of which it is desired to measure, while the smaller end of the tube is drawn down to capillary dimensions and sealed. To protect the spiral and to promote the accuracy of the instrument it may be enclosed in a vessel which can be more or less exhausted and maintained at constant pressure. Changes of pressure within the spiral cause a movement of the capillary end of the spiral, and this displacement may be magnified by a stylus or a mirror.

[1] Verh. d. d. phys. Ges., 8, 20 (1906).
[2] Z. phys. Chem., 61, 4, 457 (1908).

The sensitiveness of the instrument may be considerably increased by using glass tubing with thinner wall, by increasing the number of turns in the spiral and by increasing the diameter of the turns.

Another manometer, which likewise depends upon the mechanical effect produced by changes in pressure, has been devised by Scheeel and Heuse.[3] This instrument consists of two chambers, separated by a copper membrane. One of the chambers is maintained at constant pressure, while the other is connected with the system which is under observation. The deformation of the membrane under the pressure difference is measured by Fizeau interference bands.

Hogg[4] has shown that a vibrating disc serves admirably as a manometer for the measurement of low pressures, and that the decrement is connected with the pressure by a simple equation for pressures below 0.1 mm. down to 0.000016 mm. when the gas in the apparatus is hydrogen.

Dewar[5] has employed Crookes radiometers in the study of very low pressures, and more recently Knudsen[6] has made use of radiometoric forces in developing an absolute manometer which has been shown to be very reliable at pressures of the order of 0.0001 mm.

Quite different in principle is the manometer suggested by Pirani.[7] This instrument depends for its ability to measure pressures upon the fact that at low pressures the heat conductivity of gases is a function of the pressure. It is only necessary, therefore, to observe the effects of varying heat dissipation, with pressure changes, upon some source of heat within the vessel undergoing evacuation. Pirani has accomplished this by connecting to the system an ordinary tantalum lamp. The wire in this lamp was heated by a constant-voltage current. As the pressure in the system was decreased more and more, the heat lost by conduction through the gas was continually decreased; consequently the temperature of the wire mounted steadily, and, with the temperature, the resistance of the wire increased, thereby cutting down the current. One had then only to calibrate ammeter

[3] Ber. d. d. phys. Ges., 1909, 1-5.
[4] Phil. Mag., **19**, 376-390 (1910).
[5] Proc. Roy. Soc., A **81**, 280-286 (1908).
[6] Ann. d. Phys. IV, 32, 809 (1910).
[7] Ber. d. d. phys. Ges., 1906, 686.

readings against the indications of some form of manometer. A better instrument was obtained when the tantalum wire was replaced by platinum, and the wire was clamped tightly to the anchor wires in order to keep constant the heat loss through the supports. With the improved instrument two other methods were used in following the changes in energy loss by conduction through the gas. In order to control the resistance, and consequently the temperature, of the wire, the platinum manometer-lamp was connected into a Wheatstone bridge. One could then follow changes in energy loss by either of two methods.

(a) The wire could be maintained at a constant temperature, and the energy required at different pressures could be recorded.

(b) The current in the wire could be maintained constant, and record could be made of its resistance at different gas pressures.

In order to compensate for the effect of changes in room temperature upon the resistance of the manometer, Pirani suggested the use of a duplicate of the instrument which could be exhausted and sealed at a pressure in the region where most accurate measurements were required. This duplicate was then substituted for the resistance coil in the arm of the bridge in series with the manometer.

In simplicity both of construction and of operation, involving only current and resistance measurements within the range of instruments readily accessible, the Pirani manometer recommends itself at once. However, in point of sensitiveness to pressure changes it is somewhat disappointing. With the most sensitive arrangement described in Pirani's paper, it would scarcely be possible to follow, with accuracy, pressures lower than 0.0002 mm. or possibly 0.0001 mm., since the change in pressure from 0.0014 mm. to 0.00007 mm., as registered by a McLeod manometer, gave a change of only six divisions upon the chosen scale.

However, certain theoretical considerations led the writer of this paper to believe that the sensitiveness of the resistance manometer could be considerably increased. It is obvious that the apparent sensitiveness, *i. e.*, the change of resistance for a given change in pressure, could be increased simply by increasing the total resistance of the manometer, without in any way altering the real sensitiveness. A little reflection will make it clear that,

in comparing manometers as to sensitiveness, one must take account not only of the number of units change in resistance for each unit change of pressure, but also the relation that this change bears to the total resistance of the manometer. For instance, one of the manometers constructed in the course of experiments in this laboratory was found to have a total resistance of 264 ohms. A change of pressure amounting to 0.001 mm. of mercury caused a resistance change of 2.73 ohms in this manometer, while in another manometer, whose resistance was 129 ohms, the change was 1.92 ohms for the same change of pressure. Expressed in percentage of the total resistance, these changes amount to 0.78 percent and 1.13 percent per 0.001 mm change in pressure, and the latter manometer would be considered the more sensitive in the meaning of the foregoing definition.

It will be clear that the sensitiveness of the manometer, since its indications of pressure variation are dependent upon the changes in dissipation of heat by conduction through the gas, will be affected by conditions that will tend to make this dissipation of heat larger in comparison with the heat loss by direct radiation and by conduction along the supports and anchors. Thus the relation of the gas conduction loss to the loss of heat by direct radiation will be different, according to the temperature reached by the wire of the manometer. Also the relative amounts of heat lost by conduction through the gas and by conduction through the anchors employed to hold the wire in place will be subject to variation with different sizes of anchors and anchors of different materials, as well as their number. The heat loss by conduction through the gas may of course be increased by conditions which would give a steeper temperature gradient between the wire of the manometer and the enclosing bulb. This steepening of the temperature gradient may be brought about by (A) increasing the difference in temperature between the wire and the bulb; (B) decreasing the distance from the wire to the bulb. Again, one would use an enclosing bulb having thin rather than thick walls, thus facilitating the dissipation of heat through the gas. As was to have been expected, the temperature coefficient of resistance of the wire used has an important effect upon the sensitiveness of the manometer.

These factors, together with questions regarding the form of the filament, whether the wire should be straight, crimped, or wound into spirals between anchors, and the most favorable size of wire, were made the subjects of careful experiments. The manometer described in the following paragraphs embodies the results of these experiments. The structure of the manometer is shown diagrammatically in Fig. 1. A piece of pure platinum wire, 0.028 mm. in diameter and 450 mm. long, is

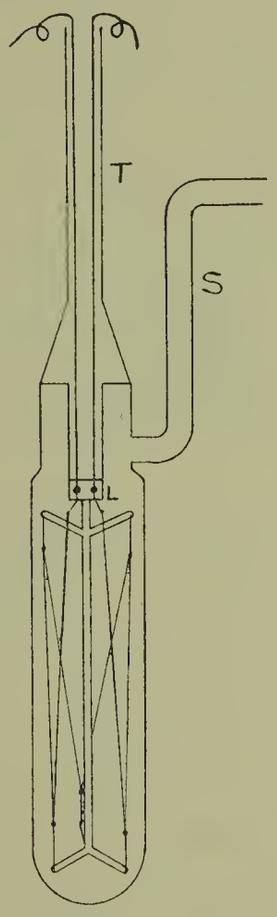

Fig. 1.

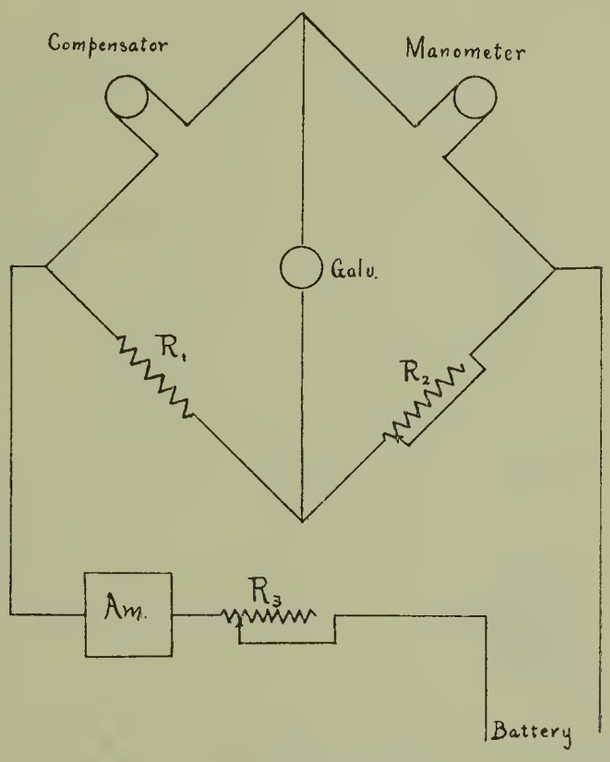

Fig. 2.

mounted upon a glass stem carrying two radial glass supports near the top and three at the bottom. The wire is anchored to these radial supports by means of short pieces of platinum wire 0.052 mm. in diameter. The anchor is fused into the radial supports at one end, and the other end is made fast to the manometer wire either by an arc weld or by a tiny glass bead. The leading-in wires at L, to which the ends of the manometer wire are welded, are of platinum, 0.31 mm. in diameter. All of the platinum wire employed in making the manometer was drawn from the same lot of larger wire and was assumed to be

of uniform purity. The temperature coefficient of the manometer wire was found to be 0.00376 percent per degree. The platinum leading-in wires are joined to heavy copper leads (diameter 1.1 mm) by welded joints, and these joints are fused into the stem as in electric lamps. The stem is sealed into a tubular bulb 3.2 cm. in diameter and 11.4 cm. long. This size of bulb is easily obtained, since it is the size regularly used for 50-watt tubular lamps, such as are commonly employed for galvanometer illumination. At S is a tube by which the manometer is connected with the system whose pressure is being studied. The upper end of the stem T is considerably elongated to permit the complete immersion of the manometer in a constant temperature bath, whose temperature was approximately 0° C. This stem tube is made of sufficient length to leave 15 cm. of it above the level of the bath, a provision which we found to be necessary in order to avoid the condensation of atmospheric moisture upon the top of the tube and the leading-in wires during humid weather. For electrical insulation this tube is packed with purified dry asbestos wool.

The compensator is, as nearly as possible, identical with a manometer, except that the side tube is omitted. A diagram of the electrical connections used with the manometer is shown in Fig. 2. The manometer and compensator form two arms of a Wheatstone bridge, the other two arms being R_1, a well-aged coil of manganin wire, whose resistance is 925.6 ohms, and R_2, a decade plug box containing 10,000 ohms. The coil R_1 is imbedded in pure paraffine, free from acid. The galvanometer employed had a resistance of 104.8 ohms and a sensibility of 75 megohms and 0.013 microamperes per millimeter on a scale placed one meter away.

The position of the ammeter, Am, in the circuit should perhaps receive explanatory comment. The current which it is desired to control is that flowing through the branch of the bridge containing the manometer and compensator, and the natural place for the ammeter is in that branch. However, the presence of the ammeter in that part of the bridge would be a source of error on account of the variation of the resistance of the meter with changes in room temperature. On the other hand, placing the meter outside the bridge is open to the objection that the

total current is measured, and not the current flowing through the branch containing the manometer. However, the distribution of the current between the two branches of the bridge will always be the same when the bridge is balanced, so that by maintaining a constant total current through the ammeter, the current in the manometer and compensator is likewise constant. The strength of the current used in all of the experiments recorded in this paper was 0.00925 amp., which was sufficient to raise the wire in the manometer and in the compensator to about 125° at the lowest pressures. This current was drawn from a storage battery of five cells, and was controlled by the

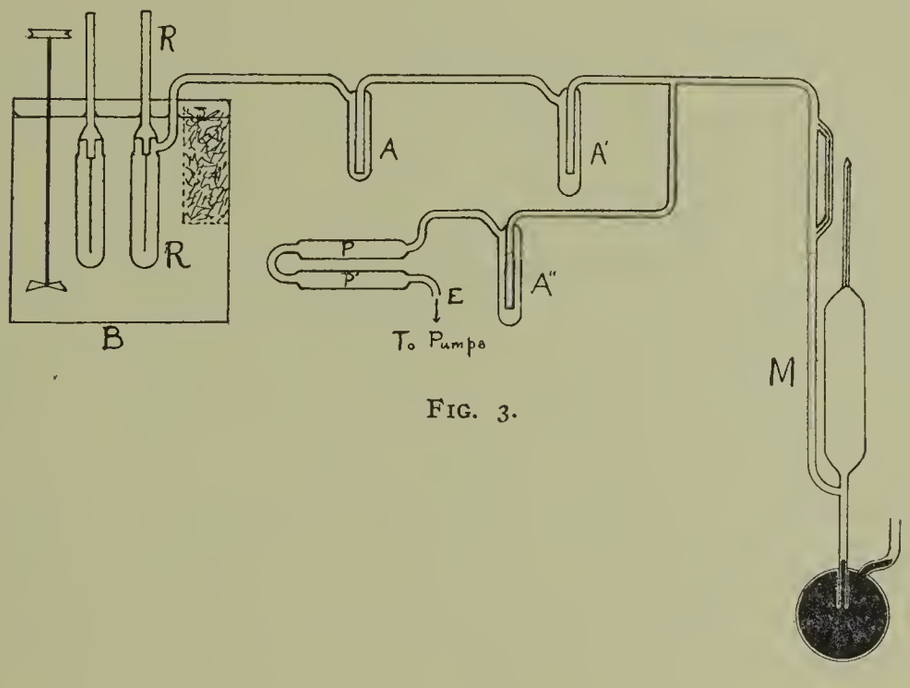

FIG. 3.

resistance R_3. The milliammeter employed was very sensitive, one scale division being equal to 0.0000667 amp.

The manometers are calibrated against a McLeod manometer, the resistance readings of the former being plotted against the pressure readings of the latter. The arrangement of apparatus for carrying out the calibration is shown diagrammatically in Fig. 3. At the right is a McLeod manometer M made of German glass. The capacity of the bulb of the manometer is 400 cc., while the diameter of the capillary is 0.7 mm. and its length 13 cm. This ratio of capillary to bulb gives a difference in level of mercury surfaces amounting to 3.4 mm. for a pressure of 0.00001 mm. in the system. At R is the resistance manometer,

about 80 cm. distant from the McLeod, the two manometers being connected by tubing whose internal diameter was 8.0 mm.

The apparatus was exhausted through E either by means of a rotary oil pump or by means of a mercury pump of the Topler type. At P and P′ are two tubes containing phosphorus pentoxide, which serve to keep the apparatus free from water vapor. The traps at A, A′ are immersed in liquid air, and prevent mercury vapor from entering the resistance manometer, and the trap at A″ prevents oil vapors from the pumps reaching the McLeod manometer.

The resistance manometer and compensator are immersed in a water bath B, which contains about 10 liters. The water is kept vigorously stirred by a propeller actuated by an air turbine. Within the tank is suspended a wire cage about 7 cm. in diameter and 20 cm. long, and this cage is kept filled with cracked ice during an experiment. This method of obtaining a relatively constant low temperature bath proved to be an easy one.

The procedure followed in preparing and calibrating a manometer will now be outlined. Compensators were prepared in duplicate by attaching two of the manometer-lamps to the exhaust apparatus, already described. They were enclosed in an electrically heated oven and baked at 360° C. for at least eight hours, the pumps being in continuous operation. Sufficient current was sent through the manometers to raise the temperature of the wire somewhat above that of the oven, although its temperature was always below red heat. Toward the end of the exhaustion, liquid air baths were adjusted to the traps (A, A′ and A″, Fig. 3), and the exhaustion continued to 0.00002 mm. by means of the mercury pump alone. The ovens were then removed and the pressure kept at that point until the compensators were cool enough to be sealed off from the apparatus.

Another manometer was now attached as shown at R (Fig. 3), and baked and exhausted for at least an hour without liquid air baths; then the oven was removed and the manometer allowed to cool. Liquid air baths were readjusted, in the order A″, A′, A, and then the ice bath was put into place. This order was adopted after we had experienced some trouble with the condensation of traces of mercury in the resistance manometer, and it was found to obviate the difficulty entirely. Probably the

trap at A is not necessary; no trace of mercury appears in it except when the liquid air baths have been in place 8 or 10 hours. The compensators were now put into the bath and the electrical connections were made in accordance with the method already described. By operating the mercury pumps or by admitting air, purified by bubbling through strong caustic potash solution, over solid caustic potash, calcium chloride and phosphorus pentoxide, the pressure in the apparatus was brought to the point at which the calibration was to begin, usually between

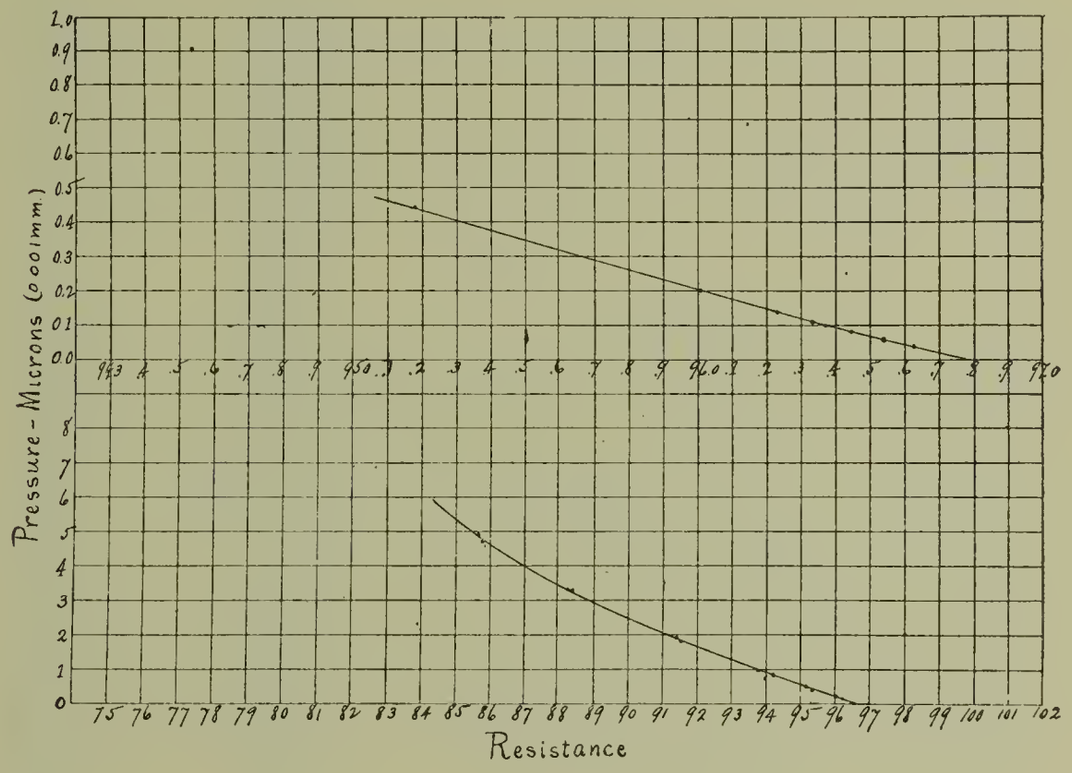

FIG. 4.

0.005 and 0.010 mm. As soon as the system had come to equilibrium, as indicated by cessation of resistance changes in the manometer, the resistance was recorded and the pressure read on the McLeod manometer. A few strokes of the mercury pump served to give a new resistance reading and a new pressure, the process being continued until a pressure between 0.00003 and 0.00001 mm. was reached. The resistance readings obtained are of course not the actual resistances of the manometer, for the reason that the ratio arms of the bridge are not exactly 100 : 1000, but about 90 to 925, and the ratio varies somewhat, of course, with different compensators. However, it is not of importance

to obtain actual resistances, since the indications of the manometer were always observed with the same electrical system.

TABLE I.

Manometer No.	Date	Pressure Mm.	Resistance Ohms
20	July 25, 1910	0.0069	82.68
		0.0047	85.73
		0.0032	88.37
		0.0019	91.37
		0.00095	93.75
		0.00044	95.28
		0.00014	96.23
		0.00008	96.44
	July 26, 1910	0.0049	85.66
		0.0033	88.33
		0.0018	91.48
		0.00080	94.17
		0.00047	95.18
		0.00020	96.02
		0.00011	96.33
		0.00006	96.54
		0.00004	96.63
34	April 10, 1911	0.00384	78.57
		0.00159	81.46
		0.00073	82.90
		0.00024	83.77
		0.000023	84.23
		0.000003	84.28
	April 11, 1911	0.00778	74.56
		0.00331	79.08
		0.00145	81.77
		0.00068	83.01
		0.00015	84.03
		0.00005	84.26
		0.000003	84.28
42	July 29, 1911	0.00291	88.00
		0.00197	89.60
		0.00078	91.49
		0.00032	92.47
		0.00010	92.93
		0.000015	93.12
	July 31, 1911	0.00459	85.66
		0.00331	87.27
		0.00200	89.52
		0.00124	90.62
		0.00056	91.97
		0.00022	92.68
		0.00007	92.99

A few sample calibrations chosen at random from a large number of manometers are given above in Table I and are plotted in Figs. 4, 5, 6. The curves for the lower pressures, from 0.001 mm. down, are plotted on the upper half of the chart, the scale

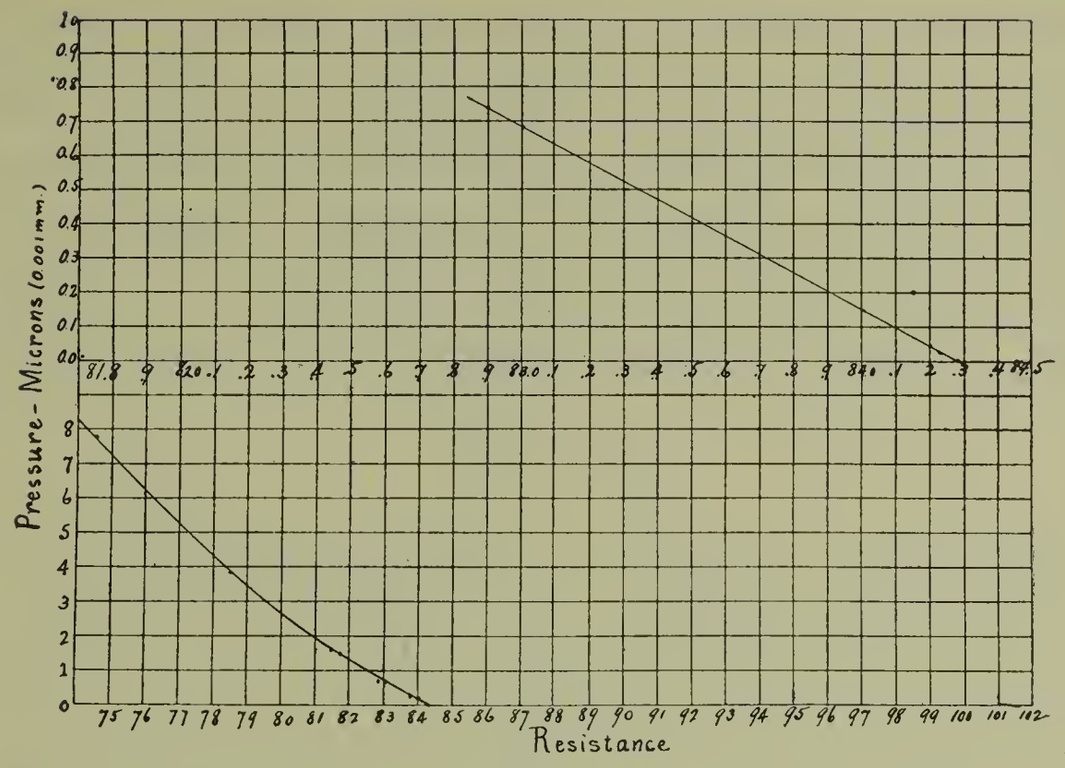

FIG. 5.

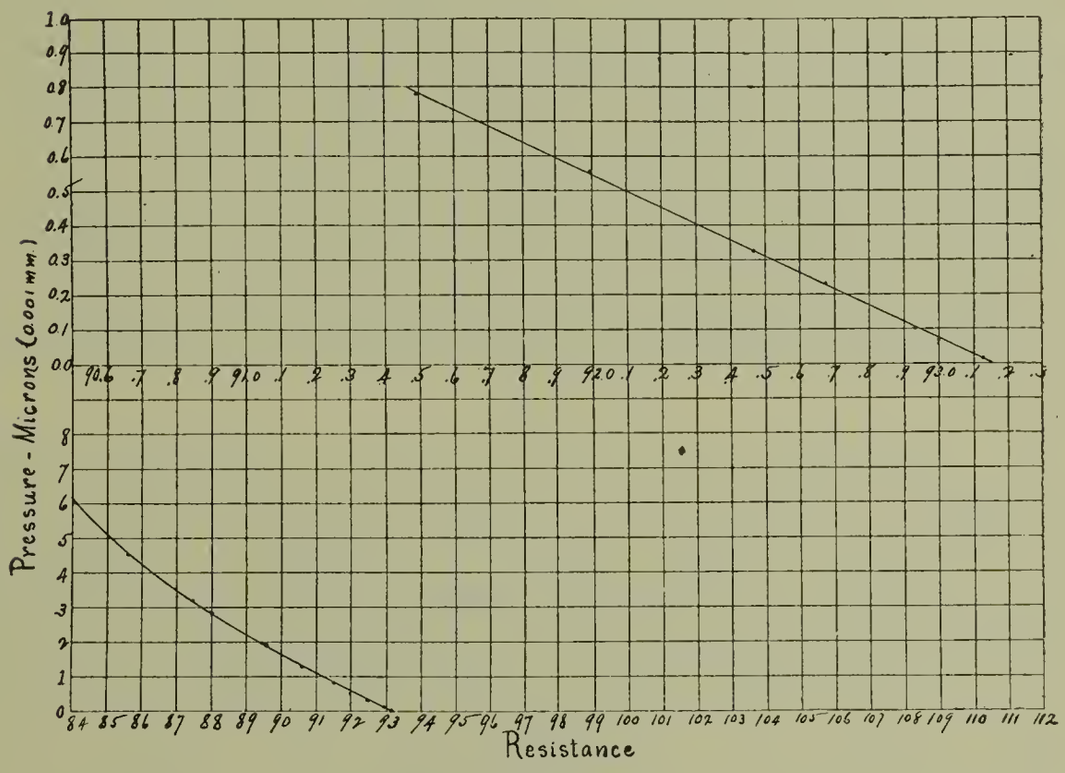

FIG. 6.

being ten times that of the lower half. The slope of the curve shows that for each change of pressure amounting to 0.00001 mm. the resistance changes more than 0.02 ohm, and as resistances can be measured to one part in 10,000 with a well-made Wheatstone bridge, it seems not unwarrantable to conclude that, with

the apparatus described, we should be able to detect and measure changes of pressure of the order 0.00001 mm. and to follow exhaustion down to a pressure of the order 0.00001 mm. The absolute values of the pressure are not obtained directly from the calibration curves, owing to the fact that the resistance manometer is at 0° C. and the McLeod manometer is at room temperature. The values may, however, be calculated in accordance with the formula derived by Knudsen,[8]

$$\frac{p_1}{p_2} = \sqrt{\frac{T_1}{T_2}}$$

Where p_1 and p_2 are the pressures, at equilibrium, in two vessels connected by a tube whose diameter is vanishingly small as compared to the mean free path of the molecules of the gas contained in the system; and T_1 and T_2 are the absolute temperatures of the two vessels. While the average temperature of the gas in the resistance manometer is probably somewhat higher than 0° C., the temperature of the bath, the extreme condition would be represented by basing a correction on 273° as the value of T_1. T_2 will of course be variable with room temperature changes, but 293 may be taken as an average value. On substituting these values, p (the actual pressure in the manometer) is found to be 0.964 of the pressure recorded by the McLeod manometer. At pressures above 0.0001 mm. this factor is of some importance, but it may be neglected at pressures lower than this.

Some observations have also been made to determine within what degree of accuracy the readings of the manometer from day to day may be relied upon. As a means of checking the system as a whole we have found the use of two compensators a good working method. The two compensators are immersed in the bath, one being connected into the bridge in the place usually occupied by a manometer, the usual current is sent through the apparatus, and when equilibrium is established, the resistance is recorded. Then the places of the two compensators are interchanged and the equilibrium resistance obtained as before. These two observations would enable us to detect any trouble in the bridge instruments and in the compensators. We have obtained readings with two compensators over a period of

[8] Ann. d. Phys., IV, 31, 205-229 (1910).

several weeks in which the maximum variation in resistance was 0.01 ohm.

Another point which has been subjected to experimental inquiry is the effect of changes in the temperature of the bath upon reading of the manometer. It is clear that when the pressure within the manometer is identical with that in the sealed compensator, variations in external temperature would have a negligible effect, but at pressures above and below that in the compensator, the compensation would be less perfect.

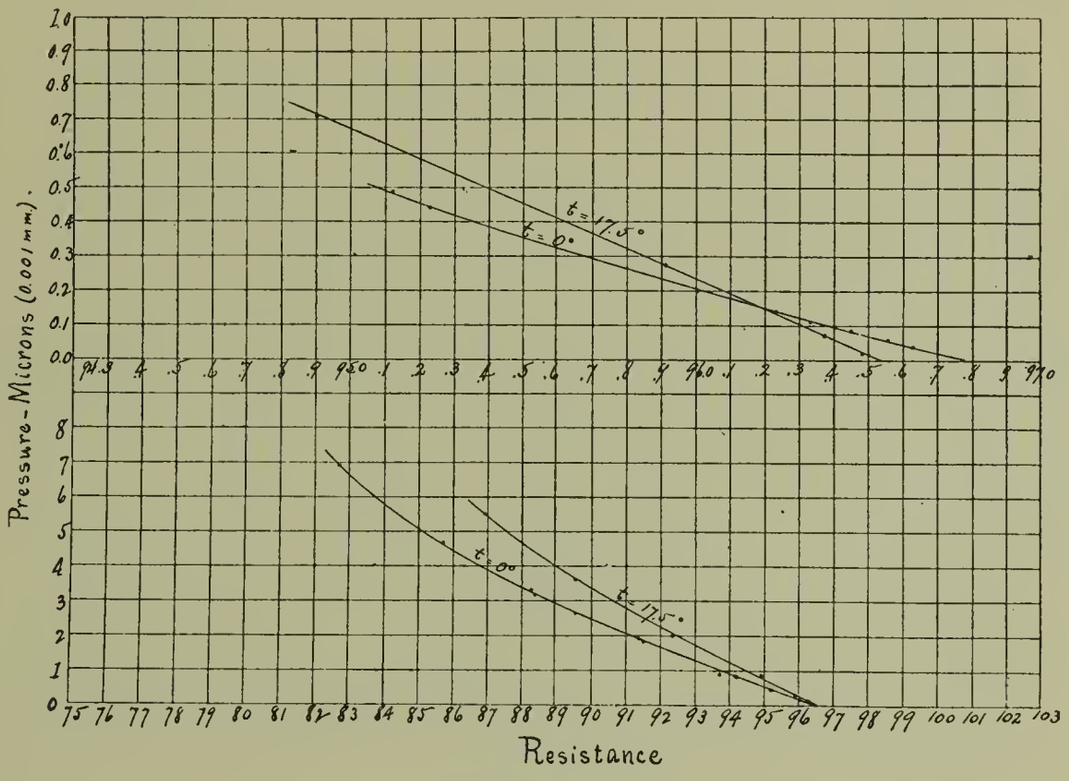

FIG. 7.

The results of an experiment in which the bath temperature was varied are given in Table II and are plotted on Fig. 7. The manometer used in this experiment was No. 20, whose calibration figures, under normal conditions, are given in Table I.

TABLE II.

Pressure Mm.	Bath Temperature 0° Resistance	Bath Temperature 17.5° Resistance
0.0055	84.59	86.98
0.0036	87.70	89.58
0.0020	91.01	92.33
0.00071	94.49	94.90
0.00027	95.80	95.91
0.00007	96.49	96.37
0.00002	96.70	96.48

It will be seen that for this particular system the compensation is perfect at a pressure of 0.00015 mm. At a pressure of 0.005 mm. the change in temperature of the bath from 0° to 17.5° increases the resistance of the manometer by 2.39 ohms, or 0.136 ohms per degree. This change of resistance would involve an error of 0.00087 mm. per degree in the reading of the manometer. At a pressure of the order 0.00001 mm. this error would be 0.00001 mm. per degree of change in bath temperatures. It has been shown that the bath may be kept constant within 0.1°

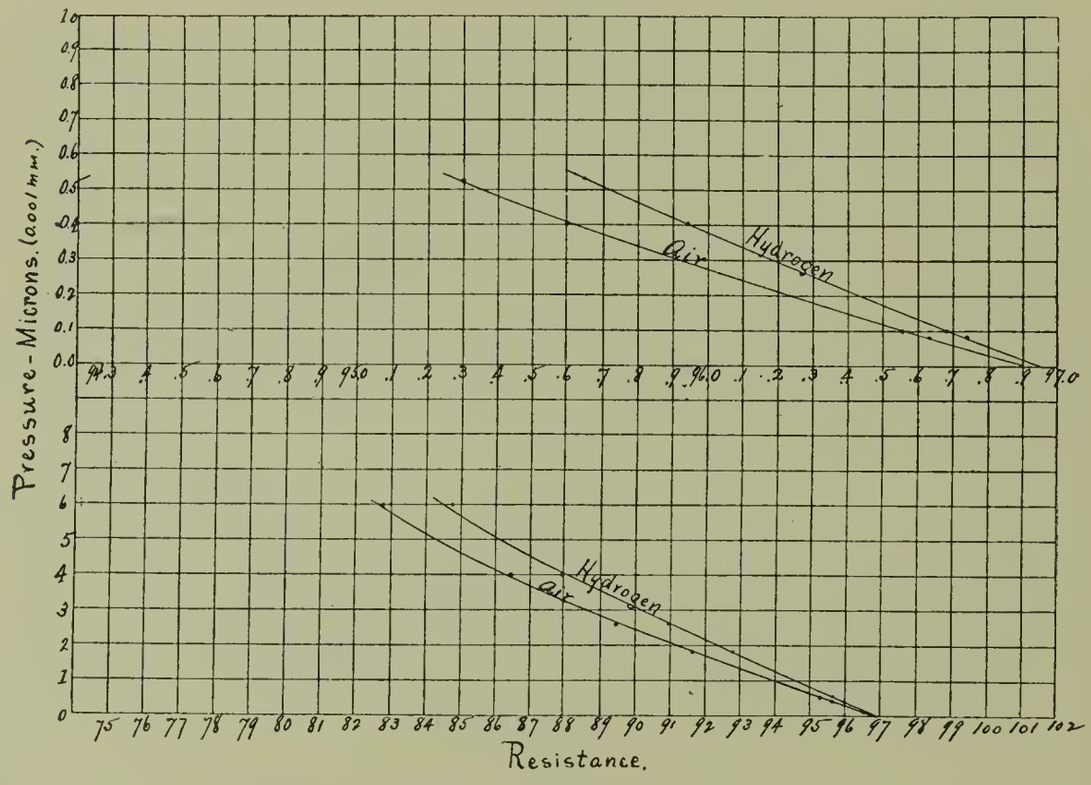

FIG. 8.

C., so that the error involved in our measurements, on account of fluctuations in bath temperature, are negligible.

One objection which may very legitimately be raised against this form of manometer arises from the fact that the heat conductivity varies with the nature of the gas so that the manometer must be calibrated with the same gas with which it is to be employed subsequently. At present we have calibrations only in pure dry air and hydrogen. In these experiments the compensator was not filled with hydrogen, so that the compensation for changes in bath temperature would be less perfect than otherwise, but this was considered a small effect as compared with the

effect of the change of gas in the system. The calibration resistances under normal conditions and after the apparatus had been carefully filled with hydrogen are given in Table III.

TABLE III.

Pressure	Resistance With Hydrogen	Resistance With Air
0.0060	82.80	84.75
0.0040	86.41	87.95
0.0026	89.43	90.95
0.0018	91.66	92.75
0.00053	95.30	95.65
0.00040	95.60	95.94
0.00010	96.56	96.68
0.00008	96.63	96.74

These values are plotted in Fig. 8. Owing to the better conductivity of the hydrogen, the resistance readings of a manometer, which has been calibrated in air, would, with pure hydrogen, lead to an error of from 20 to 25 percent of the real pressure. Experiments with other gases have not yet been carried out, but one may estimate whether the effect would be greater or less than that of hydrogen, by reference to the recent work of Soddy and Berry,[9] upon the conductivity of rarified gases.

It is hoped that further data concerning a number of phenomena in gases at very low pressures may be available for publication in the near future.

The author desires to thank Mr. A. E. Freeman, of this laboratory, for much assistance in carrying out the details of the experiments.

Summary.

The chief results of this paper may be summarized as follows:

1. The resistance manometer suggested by Pirani has been found to be susceptible of considerable improvement in regard to its sensitiveness.

2. A method of calibration of the resistance manometer has been described.

3. The manometer has been found to give reliable measurements of pressure down to 0.00001 mm. of mercury.

[9] Proc. Roy. Soc., A 83, 254-264 (1910), and A 84, 576-585 (1911).

4. The manometer must be calibrated in the same gas in which it is to be used to indicate pressures.

Research Laboratory,
General Electric Company,
Schenectady, N. Y.,
August 2, 1911.

A paper read by title at the Twentieth General Meeting of the American Electrochemical Society, in Toronto, Canada, September 23, 1911, President W. R. Whitney in the Chair.

GALVANIZING WIRE IN ZINC-DUST.

By Alfred Sang.

Ever since I presented before the American Electrochemical Society my first paper on galvanizing steel wire by heating it electrically in zinc oxide or zinc dust, I have realized that a perfectly clear conception of the forces at work was essential for successfully operating the process. Once the nature of zinc dust was well understood and the action which different temperatures may have on it was clearly demonstrated, the application of the process was found to be, after all, not nearly as complicated as might have been feared, in fact it was less complicated than the other two processes in general use.

It must be recalled that zinc dust does not melt when heated to the temperature of fusion of zinc, but at higher temperatures reducing agents will "precipitate" the metal, as it were, and it can also be squeezed out under mechanical pressure. This property of resisting fusion is supposed and is probably due to the surface of each particle being formed of zinc oxide or zinc carbonate.

If zinc dust is heated evenly, the temperature is found to rise steadily until around 390° C. when the rise becomes much more rapid, attaining its maximum rate at about 420°, then falling gradually until in the neighborhood of 470° it again becomes normal. When the dust is cooled down an opposite effect is noted between 450° and 260°. If the operations of alternately heating and cooling the same dust are repeated, the exothermic and endothermic effects are manifested each time between the same temperatures, with their apices at about 420° on the rise and about 350° on the fall.

We cannot help but notice that the exothermic effect on the rise occurs at the melting point of zinc metal, but when zinc is melted the effect is the very reverse, heat being absorbed at the moment of fusion to the extent of 28 Calories per kilogram.

We are therefore in presence of an anomalous critical point, and I will now consider separately the operations of galvanizing in zinc dust below and above that critical point.

If a clean piece of steel wire is immersed in zinc dust which is heated to a temperature below 420°, or at what is known as "sherardizing temperatures," a slow cementation takes place and zinc is absorbed from the very moment that the wire reaches the temperature of the dust; there is at the same time a slow sublimation of zinc at the surface increasing with time but sufficient for all practical purposes after a few hours of treatment; in the presence of reducing gases the time of treatment can be considerably lessened but the penetration of zinc is of course less. This process is sherardizing, unsurpassed for many purposes, unsuitable for many others. There are two varieties of sherardized coatings: the gray, porous and brittle variety, mostly composed of zinc carbonate which darkens with age; other variety has a high metallic luster and is compact and of a finely crystalline structure. If this were the class of sherardizing produced by all existing plants, there would not be much hot-galvanized work produced outside of wire and sheets.

When we work with the dust at a temperature above the critical 420°, results are entirely different; galvanizing is *instantaneous,* taking place the very instant that the wire reaches the temperature of the dust. There is very little penetration of zinc in the iron; there may be vaporization and condensation of zinc as in sherardizing, altho I doubt it. A wire heated to 800 or 900° in a non-oxidizing flame and quickly thrust into cold zinc dust will condense around itself a considerable thickness of zinc. The best working temperature ranges between 500 and 600° C. The galvanizing is bright and more pleasing in appearance than that obtained by any other means.

I will not go into details of the furnace used, which merely consists of a zinc-dust trough between two sand troughs provided with a set of cast-iron cover-plates over the zinc trough and power-driven rolls. As high a speed as 850 feet per minute can be used for securing good merchant galvanizing. It is found preferable to run at 35 feet only, running a large number of strands and attaching the coils end to end.

The process is economical as regards current consumption

which, outside of zinc and labor with a very small amount of power for driving the rolls, represents the bulk of direct cost. Figures are readily arrived at, for as soon as the dust has attained a fairly constant temperature, as it very rapidly does, the consumption of electricity in watt-hours is represented by the formula

$$\text{Watt-hours} = \frac{w \times s \times t \times 7}{6}$$

where

w = weight of wire treated.....say 1,000 kilograms.
s = specific heat of steel.....say 0.18 at 550°.
t = temperature to be maintained.....say 550° C.
w s t = calories needed, which, multiplied by the ratio 7/6, gives the watt-hours.

Figuring 75 percent efficiency for the dynamo or transformer supplying the current, the above formula gives 154 kilowatt-hours per long ton of wire of any diameter.

While the wire is softened by the process, a full annealing can only be secured by using a higher temperature, the consumption of electrical energy being then increased from 40 to 50 percent and the sand trough through which the wire passes after leaving the zinc dust must be appreciably lengthened. If a slight scale is to be removed, the high temperature must be used in connection with a reducing agent. Sand must be used with the zinc dust to prevent caking and cavitation in the path of the wire. Aluminium contact rolls and special mercury contacts are the only ones suitable for this kind of plant.

The wire can be brought out into the air immediately after coating, but it is preferable to let it cool by passing it through sand which brightens it at the same time.

The instantaneous galvanizing of steel in zinc dust above 420° is readily explained by completing the theory which I developed as far back as 1907; I will now state it in its complete form.

Zinc dust is produced by the sudden cooling of the vapor of zinc and the transition from the gaseous to the solid state is so rapid that, for all practical purposes, the liquid state is skipped. The setting of the zinc, brought about by sudden chilling, prevents regular arrangement of the molecules along determined

stereochemical lines; it forbids the formation of crystals for which time is required. The heat of solution of matter in an amorphous state is always higher than when in a crystalline state; the same is true of a strained condition which likewise presents an amorphous appearance. At the time of formation, considerable heat is retained which would have been radiated by slow cooling. The condition is one of physical instability resulting in readier and, oftentimes, explosive decomposition.

The physical instability of zinc dust may be explained thus: in the prolongs of zinc furnaces the uncondensed vapor is suddenly chilled and collects in minute drops which are instantly congealed at the surface and oxidized, forming a rigid spherical skin or crust. Within this crust the liquid zinc cools and contracts more slowly but the rigidity of the crust does not permit the drawing in of the surface to form the facets of a crystal and compensate this internal contraction. Thus, voids must exist within the crust and we may presume a structure which might be described as "piped radially" or as negatively crystalline.

Below the melting point of zinc this condition persists but affinities and thermal inducements find a readier response from zinc dust than from any other form of zinc, both by reason of the enormous amount of free surface and because of this unstable condition.

At about 415°, which is the melting point of zinc, the strain within the zinc dust particle is totally or almost totally relieved as indicated by the loss of heat, altho some of the latent heat must be retained for fusion.

At high temperatures zinc dust is pasty, because formed of innumerable little bags of melted zinc, but these will not coalesce on account of their small size and closely packed condition, unless considerable pressure is used. These minute bladders might be compared to soft-shell turtle's eggs; they form a plastic and cellular mass.

If, however, a reducing agent, say a drop of coal tar, is introduced, reduction of the skin takes place and we get a small mass of more or less spongy metallic zinc. If a metal which is electronegative to zinc, such as iron, is put into contact with these bags of zinc, reduction and an exchange of metals take place,

the bag is punctured at the point of contact and the zinc spreads itself on the iron.

Hence it is that wire can be galvanized in this manner by means of a plant closely resembling a hot galvanizing plant, for the process is, in fact, one of hot galvanizing.

A paper presented at the Twentieth General Meeting of the American Electrochemical Society, in Toronto, Canada, September 21, 1911, President W. R. Whitney in the Chair.

TITANIUM IN IRON AND STEEL.

By Charles V. Slocum.

In electrometallurgy no development of late years has been of greater importance than the manufacture of ferro-titanium in the electric furnace. It was not until 1903 that Auguste J. Rossi was able to produce the remarkable material now widely known as "Titanium Alloy." Some thirty years or more of his life has been devoted to developing the methods of manufacture and the uses of this material, but it was not until he used the electric furnace in 1903 that his life-long efforts were crowned with success. Since that time the manufacture and consumption has grown with leaps and bounds, and now the plant occupies a large tract of land, and railway facilities are necessary for the prompt handling of the large output.

As the uses of this alloy are so many and so varied, it has been suggested by a well-known member of this Society that the more important features be discussed in a suitable paper. Metallurgists have practically all agreed that the use of a small percentage of an alloy of titanium is of benefit in both iron and steel, but some question has arisen as to the form which this alloy should take. In a recent publication[1] the statement has been made that carbon-free metals dissolve more easily than metals containing carbon, since the latter contain carbides. The experience of the writer does not bear out this fact, he having found that a carbon-free alloy was not at all adapted to use in the iron and steel industry, and the manufacturers were obliged to make a product containing from 5 to 8 percent carbon for such use. Our early experience with an alloy of iron and titanium free from carbon, but containing 5 to 10 percent aluminium, resulted in almost complete failure, as the aluminium when present in any

[1] Goldschmidt: The Melting Point and its Relation to Alloying Capacity. Met. & Chem. Eng., 9, 348 (1911).

appreciable quantity made the steel brittle and its oxide showed great tendency to remain in the steel.

"Titanium Alloy," as stated above, contains 5 to 8 percent of carbon, mostly in the form of graphite. Analyses of this material made in the laboratory of Dr. C. F. McKenna show the following proportions of the two forms of carbon:

Sample No.	Graphitic Carbon	Combined Carbon
141	9.601	0.147
162	9.179	0.12
291	7.012	0.13
298	6.234	0.118

From these analyses it appears that titanium acts very much like silicon in that it causes a separation of carbon as graphite. Dr. G. B. Waterhouse recently cited an interesting experiment to bear out this fact: A ladle of iron for making malleable castings was treated with 10 percent titanium alloy sufficient to equal 0.1 percent metallic titanium added to the iron. The original iron gave castings showing a white fracture with a little graphite in the center. The titanium-treated metal showed a white border between ⅜ and ½ inch (1 to 1.25 cm.) deep and a gray center showing the separation of graphite. Contrary, therefore, to the viewpoint of Dr. Goldschmidt, there need be no fear of the non-solution of the electric furnace titanium alloy, as it does not contain more than the merest fraction of the unreadily soluble carbide.

The density of a ferro-alloy to be used as ferro-titanium is used has an important bearing on its incorporation in the molten bath. Careful determinations made on the electric furnace product containing both 10 and 15 percent titanium show a density of 6.20 to 6.40. The metallothermically produced alloy shows a density of between 6.20 and 6.30. The difference between the densities of the two alloys is therefore hardly noticeable.

The alloy is best added to the ladle of steel after tapping from the furnace and before the slag begins to run. For open-hearth steel the supply should be placed convenient to the ladle and shovelled in precisely as so much coal. For soft steel an addition equivalent to 0.03 percent titanium is sufficient to make the steel more ductile. Larger quantities are of increased benefit, but can-

not always be added because of the carbon content and also because of the density imparted to the steel, which increases as the proportion of alloy is increased, and therefore is not always desirable. In high-carbon steels this increased density is desired and more alloy is used. The addition of the alloy permits the use of a considerably higher carbon content without increasing the brittleness, and several railroads are now using as much as an equivalent of 0.10 percent titanium or more in securing tough, durable rails.

In the crucible practice the best results have been obtained by adding the titanium alloy to the pots before they are removed from the furnace, giving more time for washing and deoxidizing before teeming.

Leading authorities seem to agree that titanium achieves its remarkable results through its strong deoxidizing powers together with its effect of giving the slag formed sufficient fluidity to completely separate it from the metal.

"The presence of titanium oxide lowers the melting point of slags occluded in iron and steel and imparts thereto sufficient fluidity to account for their elimination."[2]

"Titanium * * * has a stronger affinity for oxygen than have the other well-known deoxidizers; * * * it probably gives the slag such a consistency that it separates more completely from the molten iron."[3]

"The treatment of all steels with ferro-titanium for the purpose of purifying the metal is strongly recommended, the presence of nitrogen to the extent of .02 to .035 or .045 in certain steels being enough to cause the metal to break asunder, destroying all elongation and reduction of area."[4]

"The great affinity of oxygen and titanium is an absolutely sure means of completely deoxidizing the steel, the advantages of which need no further elaboration. It has been proved that an ingot of steel containing a very small quantity of titanium will show practically no segregation."[5]

The cost of treatment with titanium varies from a minimum of 25 cents to a maximum of $2.00 per ton of metal treated. It is

[2] Bradley Stoughton, U. S. Patent Office Proceedings, Ser. No. 463610.
[3] Henry M. Howe. Ibid.
[4] H. le Chatellier. Paper, Congress of Metallurgists, Belgium, 1905.
[5] E. von Maltitz. Stahl und Eisen. No. 41. 1909.

the cheapest deoxidizer above the grade of manganese or silicon, and a far greater purifier than any other alloy. The amount to be used is very small, and should be proportioned according to the carbon content of the steel and to the amount of impurities present. To the electrometallurgist it is a triumph worthy of record that Rossi should take an element like titanium, so long considered useless, and make out of it so wonderful a servant in the iron and steel industry.

DISCUSSION.

DR. H. GOLDSCHMIDT (*Communicated*): Mr. Slocum claims in his paper that a carbon-free ferro-titanium alloy is almost useless for iron and steel. This statement refers, undoubtedly, to the carbon-free alloy of titanium which is sold in the United States by the Goldschmidt Thermit Company of New York. But this claim of Mr. Slocum is absolutely contradicted by the undisputed practical results of a large number of prominent metallurgical works which continually use the carbon-free ferro-titanium.

I do not claim that the alloy containing carbon, which has been placed on the market, is unsuitable or useless. But, on the basis of my experience, I am convinced that the ferro-titanium containing aluminum, which is made by my works, is a far better and more effective agent than the low percentage ferro-titanium containing carbon. The content of aluminum in my alloy is essential.

The experience of many years has shown that ferro-titanium which contains no aluminum, or only very small quantities, alloys with the steel only with difficulty.

In 1897 I placed on the market a 35 to 40 percent ferro-titanium containing very little aluminum, but it did not find a ready sale, since it was found exceedingly difficult to alloy it uniformly with the molten steel. The manager of an open-hearth steel plant to which I had furnished an alloy of especially high percentage inquired whether I could not make the product at a low cost, as they were desirous of using this ferro-titanium

as an open-hearth furnace lining, so little was it affected by the molten steel. I was therefore forced to place a low percentage alloy on the market. But neither was this as successful as I had hoped. Only after I added about 2 to 4 percent aluminum, so that the alloy contained one part of aluminum per three or four parts of titanium, the steel works seemed satisfied with the product. While ferro-titanium was formerly with me a product of secondary importance, it is now one of the chief products of my works. This proves that ferro-titanium in the form now made by me produces the effect desired by the steel works.

The addition of aluminum has a three-fold purpose. As mentioned before, experience has shown that the titanium alloys better with the steel bath if a certain, not too small, percentage of aluminum is present.

Secondly, the titanic acid, which is the main product formed when titanium is introduced into the iron or steel, is very difficult to fuse; it is therefore not easy to remove the titanic acid which is set free in solid form in the steel bath, and remains, partly in suspension in a finely sub-divided condition. This can be proven by microphotographic methods. This is the same property which aluminum also has, forming alumina difficult of fusion. The enclosures of the little red crystals of Ti_8CN_4 can be seen with the microscope.

It is different, however, if an alloy of aluminum and titanium together is introduced into the steel bath; in this case titanate of aluminum is formed, which melts much more easily than titanic acid or alumina alone. A simple experiment will prove this. Let us try to melt either alumina or titanic acid separately by the oxy-acetylene flame in a shallow crucible or on a refractory brick containing a depression. Only after a prolonged action of the oxy-acetylene flame, very small globules of titanic acid on one side and of alumina on the other will be obtained.

If, however, we mix the oxides in the proportion of 3 to 4 parts titanic acid and 1 part alumina—which proportion results when the 24 per cent alloy with aluminum, and free from carbon, is added to fluid iron or steel—we soon melt the mixture of titanium-aluminum oxides.

A similar method, as I see it, to this method of changing the

not easily melted titanic acid to a fluid slag has been used for a long time when adding aluminum as a deoxidizing agent to steel, that is, one alloys it with silicon and manganese.

This product is produced under a patented process by Krupp, and it has been on the market for years as the so-called S. A. M. metal (German patent No. 6332, 1895).

Further, the aluminum serves its purpose of cleaning and deoxidizing before the titanium is brought into action, so that the titanium can exert its full power, and is not called upon to do the work of deoxidizing, which the aluminum is better able to do.

For this particular reason the ferro-titanium containing aluminum is more serviceable and far-acting than that which contains no aluminum. This result has been proved by many concerns who have made experiments with both products.

As to Mr. Slocum's statement that titanium is a stronger deoxidizing agent than the others used in practice, and his reference to Dr. Howe in support of his statement, I must take exception. Aluminum is a stronger deoxidizing agent than titanium, for aluminum will reduce titanic acid.

On the other hand, I quite agree with Mr. Slocum that titanium, as shown by past experiments and practice, is a good purifier, and stands in the first rank as an agent for the production of a dense, non-porous steel. Besides acting as a deoxidizer and for removing nitrogen, the titanium itself undoubtedly has some beneficial effect on steel and cast iron.

In the cleansing of the metal bath the great affinity of the added metal for oxygen and its speed of reaction are important, but it is undoubtedly the specific properties of the added metal itself which help the result. For example, metallic calcium has a greater affinity for oxygen than aluminum, but, in spite of this, though many attempts have been made, calcium is not generally used in steel deoxidation.

The alloy of calcium and silicon which I have produced by my patented process (as a substitute for aluminum), and is now on the market, has not shown the results expected and hoped for, in spite of the fact that this alloy is decidedly a good deoxidizer. Practical results with this alloy have not equaled those of

aluminum alone and what, to a far greater extent, the aluminum-titanium alloy has accomplished.

I have always followed with great interest Mr. Rossi's work in the titanium field. He has worked hard to get on the market a high percent ferro-titanium. He had finally had to choose a product running 10 to 16 percent titanium, with a high carbon content, in order to make its introduction into steel possible. It is perfectly apparent that a low percent product, over-ballasted as it is, is not as desirable as a high percent alloy. I do not, however, in any way want to minimize the services of Mr. Rossi, as I appreciate all the difficulties he has overcome in producing the results he has.

My work in connection with ferro-titanium, as shown by my record, began in February, 1895. Somewhat later, after several hundred melts had been made and analyzed, I delivered the material to works on the Continent and in England, and some of these, while not all large buyers, are still steady customers. These remarks refer to a product that contained 20 to 25 percent titanium and a very small percentage of aluminum that was hardly noticeable.

In order to introduce small quantities of titanium into cast iron, I have, for the last six or seven years, furnished the so-called titanium thermit. This is a thermit which, when introduced into iron or steel, liberates a fluid alloy of ferro-titanium, while the slag rises to the surface. By this addition of ferro-titanium a double object is accomplished.

First, a small amount of titanium is added to the bath, and, second, owing to the fact that the can is attached to the rod during the reaction of the titanium thermit, the bath is for several minutes violently stirred, thus permitting a quick mechanical separation of the slag.

Cast iron, for this is the product usually treated, owing to its cleansing action, shows up brighter and more transparent in color, proving that a rise in temperature has taken place. Actually, as a calorific calculation will show, this rise in temperature is very small, for in a ladle of several tons a box containing only several pounds of titanium thermit is sufficient, therefore the actual rise in temperature is hardly measurable. The quality

of the cast iron is, however, decidedly improved by the addition of the titanium and the mechanical stirring.

I refer to this use of titanium in the so-called titanium thermit to prove that *I also* have devoted myself to the metallurgy of titanium for some years, and, I might add, not entirely without results. I may, very possibly, have occasion to make some further remarks on titanium when some work that I now am engaged on is completed.

Dr. R. Moldenke (*Communicated*): My experience with the element would indicate that its chief value, and possibly its only value, is as a powerful deoxidizer for iron, copper and probably a string of other metals which do not enter very much into our scheme of foundry economy, and hence have not been experimented with. We are beginning to realize more and more that the occlusion of gases and solution of oxides of metals in these metals, from the faintest traces to determinable quantities, have the effect of masking the real characteristics of the material to such an extent that we will soon find oxygen determinations part of regular laboratory returns.

The probability is that these occurrences lead to a disturbed molecular structure on the part of iron, etc., or to a serious diminution of the cohesive power of the crystalline aggregate, and hence to failure in service long before the chemical composition would warrant it. In other words, the ordinary laboratory methods entirely fail to take cognizance of this feature, primarily due to imperfect melting or annealing practice.

These oxidation effects are not only the result of a remelt under oxidizing conditions—as, for instance, in the cupola or the shaft furnace—but actually occur in the blast furnace, where there is supposedly no chance for oxidation whatever. As a matter of fact, nothing is so sensitive to oxidation as the iron sponge as it passes down from the zone of reduction to that of smelting. It requires but to get near that region of the tuyeres, where there is still some free oxygen present, to get re-oxidized again, and then the damage is done. From the greatest freedom from oxidation in the case of the cold-blast charcoal furnace, to the greatest chance for oxidation in the high-pressure hot-blast coke furnace, with a production forced to extremes, to my

mind we have the real cause of the great differences in strength of pig irons of the same shape, composition, and made under the same casting conditions.

Now comes along something that we can add to molten metal to remove this oxidation—which in the worst cases probably does not go over 0.08 in cast steel or iron. Titanium, by its practically instantaneous action, as its iron alloy is mixed with and rabbled into a ladleful of molten iron, serves to cleanse the metal from occluded gases by uniting with them, and to break up the oxides by reduction by the titanium in the alloy added. The result is giving the metal a chance to show what it can stand physically—as the result of its composition—without having the issue clouded by constituents not shown by the ordinary analysis.

If the above is true, it would seem that there is a given proportion of titanium which, when added properly, will do its work, and, if more is used, no special good will result. If, on the other hand, too little has been added, the deoxidation is incomplete. This is actually the case, and I have found that with, say, one-twentieth of one percent titanium added to a given iron, a small increase of strength being noted, twice that amount gave a marked improvement, while again doubling the amount made no appreciable difference. In other words, it was not the virtue of a titanium addition—the titanium being intended to remain in the casting, as might be wanted with nickel-steel, high manganese cast iron, etc.—that counted, but simply the freeing of the treated metal from influences that should not have been present in the first place.

There follows another consequence from the above, and that is that metals would require additions of titanium in proportion to the amount of oxidation existing in them. A case in point may be cited in the recent elaborate tests with titanium in "malleable." Here considerable additions of titanium gave but very slight indications of improvement. The real reason for this was not that the titanium did not cleanse, but that there was nothing to be cleansed out of the metal. The "malleable"—made in the open hearth—was of first-class quality. Failure to obtain results with titanium have been so frequent not because titanium is not a first-class agent, but because the metal could not be improved except by changing its composition radically.

From my very first tests with alloy additions to the melting furnace I have always avoided exposing them to the oxidizing influences of the hot gases. I would never advocate adding ferro-manganese, silicon, titanium or any other very high grade alloy to the cupola direct. In melting in little globules there is such a loss of the easily oxidizable element involved that it is deliberately throwing money away. Thus, if one wants to get the benefit of silicon in the cupola direct, it would be by using a 5 percent silicon pig in the mixture as the maximum economical percentage. For manganese this drops down to a 2 percent manganese pig iron. As the sensitiveness of an element to oxygen goes up—notably with aluminum, titanium, vanadium, etc.—the percentage of the particular element in the alloy should go down, and hence I cannot feel that it is right to use a titanium pig iron with very much titanium in it directly in the cupola, unless one does not care about expense. When pig irons with high titanium percentages—as things go—are made regularly in the blast furnace, and in this way countless millions of tons of ore become available, a new era for good iron will be at hand.

In cupola practice I therefore prefer to add a titanium alloy to the ladle of molten iron. If the alloy is in lumps, these are better made red hot before adding. Other alloys, which are brittle enough to powder, I find easier to use by sprinkling on the stream of molten metal flowing from the cupola spout. Before the stream gets down into the ladle, the alloy will have become red hot and partially dissolved. The boiling action, if any, or thorough stirring incorporates it thoroughly.

In the case of the air furnace, or the open hearth, the alloy is added preferably just before tapping. Where the slag is skimmed, a little had better be left on the bath, so that when the lumps of alloy are thrown in, there may be some protection for them from the oxidizing gases. Active rabbling is necessary to wash as much of the molten metal against the alloy lumps, until they are dissolved, and thus as much of the metal as possible brought into contact with the beneficial alloy. The bath must then be tapped, otherwise the oxidation soon makes its appearance.

Experience has shown that, with practically all alloys, one-tenth of one percent is the best proportion of the element involved to be added. Subsequent experience will indicate whether the

percentage should be greater or less. As stated above, where a good material is made, there is no necessity of adding titanium or any other cleansing alloy, and self-investigation is in order to see whether you are making the best of material. If the titanium gives no appreciable benefits, you may be pretty certain that, if it was used properly, you are doing pretty well; but if you find that you get 20 percent increases in your strength, you may be certain that your practice is still open to improvement. I may confess that I have made lots of castings in my time which were 20 percent stronger by reason of the addition of titanium alloy, "and there are others would find the same, if they tried."

MR. F. A. J. FITZGERALD: I am somewhat interested in this titanium work, because I have had occasion to make some experiments with titanium carbide. I was shown recently an interesting pamphlet issued by the Goldschmidt Thermit Company, presumably representing Dr. Goldschmidt's views. The pamphlet says, in part:

"The late famous French metallurgist, Henry Moisson, states, in his book, 'Le Four Electrique,' that the formula for titanium carbide is TiC, which indicates that 12 parts of carbon are combined with 48 parts of titanium, or, in other words, 1 percent of carbon in the ferro-titanium takes 4 percent of the titanium in order to form titanium carbide. A low percent of titanium carbide with 5 percent to 8 percent carbon is therefore not an iron-titanium alloy, but an alloy of titanium carbide and iron, and is in appearance very dark, almost black, in color, and closely resembles ordinary slag. Even if an alloy contains as much as 20 percent titanium and only 1 percent carbon, 4 percent of the alloy, or 20 percent of the titanium content, will be in the form of titanium carbide, so that actually the product is only equal to carbon-free alloy containing 15 to 16 percent titanium."

The argument here is an obvious *non-sequitur*. It might be argued in the same way that ferro-silicon made by the carbon process must contain carbon combined with the silicon, because a carbide SiC exists. That seems to be the argument that Dr. Goldschmidt advances against what he calls the carbon alloy. Now, when silicon is added to iron containing carbon, the tendency always is to throw the carbon out of combination so that it

exists in the free form as graphite. Actually the same thing happens in the case of titanium carbide. I have made many analyses of titanium carbide, made both by the aluminum process and the carbon process, and I find that in the material produced by the carbon process practically all the carbon present is in the form of graphite, and not in combination. Furthermore, when titanium carbide is added to molten iron at sufficiently high temperature, it is decomposed. Therefore it seems that Dr. Goldschmidt's argument is very weak. Furthermore, if titanium has such an extraordinary affinity for carbon, as Dr. Goldschmidt would make out, why, when low-carbon alloy is added to cast iron, does it not instantly form titanium carbide?

Dr. G. B. Waterhouse: Perhaps I can best help the members by giving a few results from the experience we have had in using ferro-titanium alloy. We have found absolutely no trouble at all in getting the steel to take up this alloy containing carbon, and this it does not just one day, or one week, or one month, but constantly. We have had absolutely uniform results both with regard to the composition of the alloy supplied and with regard to the results we have obtained with it.

There is one change I would like to see made in Mr. Slocum's paper. In the paragraph near the bottom of the second page, he says: "The alloy is best added to the ladle of steel after tapping from the furnace and before the slag begins to run." Now, if one word is changed, there it describes perfectly the way in which we add the alloy: "The alloy is best added to the ladle of steel *during* tapping." It is found best to add all ordinary deoxidizing alloys first, and then follow with the ferro-titanium. For instance, in making open-hearth steel, we add the ferro-manganese and ferro-silicon in the ladle, as is usually done in this country. The alloys are added after the bottom of the ladle is covered with the stream of metal, and as soon after as possible we follow on with the ferro-titanium. It is just shoveled in in the usual way of adding ferro-manganese, and, as Mr. Slocum says, care is taken to get it in before the slag comes, because if the slag stream comes with the metal, of course its low specific gravity makes it stay on the top, and it is very hard for the alloy to force its way down through the slag, and a good deal is lost. This does not apply only to ferro-titanium; it

applies also to ferro-manganese and to ferro-silicon; so that every endeavor must be made to get it into the steel, and not let it stay in the slag.

Then not alone must the alloy be incorporated with the bath of metal, but it is very essential that time must be given for the changes to take place. That is a point that is scarcely brought out strongly enough by Mr. Slocum, although it was mentioned by another. The oxides that have formed, and the various gases or compounds, whatever they are, must be given time to form. The changes, of course, in a chemical equation must have some time to take place, and in steel-making we must also give time for the products of those equations to separate from the steel and rise into the slag. That is a point I would lay special stress on. Not only must the alloy be thoroughly incorporated with the steel, but sufficient time must be given for the changes to take place.

In making Bessemer steel, with which, also, I have had experience, the method is almost the same. The spiegel mixture and ferro-manganese are added to the steel first, and then the ferro-titanium. In the plant where I work, we add the spiegel mixture to the Bessemer converter, and give it time to do its work, and then pour the steel into the ladle, and, as we are pouring it, add the ferro-titanium, and then give it time to do its work before we pour the steel into the ingots.

The method of adding in the foundry has been described by Dr. Moldenke in the communication Mr. Lidbury read.

PRESIDENT WHITNEY: I would like to ask someone present to make a rough estimate as to the amount of ferro-titanium alloy used in this country. I think it would be interesting to members who are not very familiar with the industry. I myself looked up the matter out of curiosity, knowing very little about it. I think I found that something like 200,000 tons were made in this country last year. Can anyone tell us about how much is used in general, so that we may get an idea of the industry?

MR. CHAS. V. SLOCUM: I want to go back a little, if I may, Mr. President. Mr. Robertson mentioned, in his paper on "Iron Smelting in Sweden," the use of some ore containing a fraction of one percent of titanium, if I remember his statement correctly.

I want to say in that connection that, as far back as 1892, I happened to be connected with a concern that built a furnace for Mr. Rossi to operate with titaniferous ores, and he found that there was no difficulty in smelting ore that contained as high as 16 percent TiO_2, but, contrary to the statement which was made later in one of the papers today as to the use of iron ore containing titanium, this smelting of the ore merely made the titanium pass off into the slag, and a very clean pig iron resulted which contained perhaps a trace, but no available percentage, of titanium.

Dr. Moldenke mentioned the "necessity," as he called it, I believe, of heating the alloy, making it red hot before using. I have looked after the addition of the alloy from our plant in more than a hundred cases, in Canada and in the United States, and I have never yet seen it heated, and have never yet seen it fail to act readily when used cold, as one might use ferromanganese.

On this question of the use of an alloy which contains carbon, or an alloy which contains aluminum, I have been handling, as general sales agent of the Titanium Alloy Manufacturing Company, a product which contains carbon. We made the alloy at first, and for perhaps a year, by use of aluminum, and we had difficulty in incorporating it into the steel at times, and had almost regular failures in using it in iron; but there are too many steel manufacturers and iron foundries using the carbon alloy throughout America today to make it necessary for me to comment upon that feature of the subject. It is usable, it is cheap, and it does the work. On this point, William Metcalfe, in his book, "A Manual for Steel Users," makes the statement that the steel maker must be very careful in his use of aluminum. He says he thinks it is almost unnecessary to caution him, because the addition of half an ounce at the most is sufficient to a ton of steel, and a little too much would pipe an ingot "from top to bottom." Now, if aluminum is, I might say, an embrittling element in the manufacture of steel, unless very carefully used, I assume that manufacturers would prefer to add aluminum separately, and thereby be able to gauge it accurately. In the use of the carbon alloy no such precaution is necessary. It contains, if any aluminum at all, the merest fraction of a percent. It

contains impurities guaranteed not to total over one-half of one percent when we take the iron, the carbon and the silicon separately, so that you are using an element which adds no impurities and which gives a definite quantity of carbon, which may be accurately figured and accurately used.

I want to say, in endorsement of Dr. Waterhouse's statement, that to my positive knowledge the company which he represents has used during the past two years more than 2,000,000 pounds of titanium alloy, and the Titanium Alloy Mfg. Co. has made a quantity running into the hundreds of thousands of tons. The foundry has a capacity of 80,000 to 100,000 pounds per day, and, except during the summer season, we have operated that foundry night and day.

President Whitney: I think that, to a layman, as I consider myself, it is an important point to know that one company can make 100,000 pounds a day of that alloy. It shows that it is coming very rapidly to be a business. We have also learned that the alloy is a good thing, and that there are two different ways of making it, and that both of them work.

A paper presented at the Twentieth General Meeting of the American Electrochemical Society, in Toronto, Canada, September 21, 1911, President W. R. Whitney in the Chair.

NOTE ON AN UNSUCCESSFUL FURNACE EXPERIMENT.

By Francis A. J. FitzGerald.

The experimental work which forms the motive of this note was unsuccessful in that the results obtained were not those expected. On the other hand, the observations made were instructive and are believed to be worth recording, since they may be of assistance in enabling others to avoid similar errors.

At the Nineteenth General Meeting of this Society a paper on "A New Electric Resistance Furnace"[1] was read. In this paper the design of a new form of furnace was fully described and certain figures given showing the results obtained with similar furnaces of an earlier model. Since that time experiments were tried with the furnace described in the paper, with disappointing results. For, while the working of the furnace, so far as its electrical properties were concerned, was most satisfactory, the efficiency was very low, owing to excessive heat losses. A comparative study of this furnace, and similar ones of a somewhat different construction, is instructive, and forms the subject of the present note.

The table below gives certain data in reference to the furnaces constructed on this principle, the furnace number appearing in the first column. In the second column the nominal electric capacity is given; in the third column the temperature of the hearth at which experiments were made; in the fourth the heat losses in kilowatts, determined by holding the furnace for a long period at a constant temperature and measuring the rate of generation of energy necessary to maintain that temperature constant; the fifth column gives the percentage loss referred to the electric capacity of the furnace. The columns headed "A" and "B" give certain ratios which will be explained later.

[1] These Transactions, **19**, pp. 273-284 (1911).

Furnace No.	Capacity K. W.	Temperature Cent.	Losses K. W.	Losses Percent	A	B
1	40	1300	18	45	0.20	1.1
2	40	1240	15	38	0.20	1.1
3	150	1250	33	22	0.29	1.0
3	150	1450	42	28	0.29	1.0
4	75	1100	38	51	0.20	2.0
4A	75	1200	35	47	0.22	2.0

Furnace 1 had walls 230 mm. (9 in.) thick. The resistor was "open," that is, it radiated directly on the hearth.

Furnace 2 had walls 345 mm. (13.5 in.) thick, the outer part of the wall being built of hollow bricks. The resistor was "enclosed," that is, there was a septum of refractory material interposed between the resistor and the hearth.

Furnace 3 had walls 345 mm. (13.5 in.) thick, and between these walls and the iron casing of the furnace was a space of 30 mm. (1.2 in.) filled with kieselguhr.

Furnace 4 was of the design described in the paper referred to above, and had double walls, each 230 mm. (9 in.) thick, separated from one another by an air space 25 mm. (1 in.) wide.

Furnace 4A was the same as Furnace 4, except that part of the walls was covered with a layer of heat insulating bricks 55 mm. (2.2 in.) thick.

Now, since Furnaces 4 and 4A were supposed to be the most improved model of this kind of furnace, the very high heat loss was extremely disappointing. In looking for an explanation of the poor results obtained, the first thing naturally suggested was that the septum between the resistor and the hearth might offer such a high resistance to the flow of heat that the efficiency of the furnace would be low. However, as this hypothesis was formed during the experiment, it was an easy matter to test it by the simple expedient of knocking out the septum. As this produced no appreciable improvement in the working of the furnace, the hypothesis ceased to be useful.

There then seemed to be two other explanations of the phenomenon; either the design of the furnace was radically wrong, or the loss of heat by conduction through the furnace walls was excessive.

The heat energy radiated from the resistor is absorbed by

the charge on the hearth of the furnace on the one hand, and by the walls and roof on the other. Now, the heat absorbed by the walls and conducted away to the surroundings of the furnace is lost, and, consequently, other things equal, it is desirable to keep the ratio between the hearth area and the total area receiving heat from the resistor as large as possible. This ratio has been calculated for the various furnaces and is given in the column marked "A" in the table. It will be observed that the ratio is greatest in the case of Furnace 3, and this is at any rate a partial explanation of this furnace's superiority.

Another important consideration is the ratio between the area of the surface, other than the hearth, receiving the heat radiated from the resistor and the rate of generation of energy in the resistor. This ratio has been calculated for the various furnaces, and is given in the column marked "B." Here it is seen that the ratio for Furnace 4 is twice as great as in the case of Furnace 3; consequently, other things equal, the heat losses would be twice as great..

It does not follow, however, that, because the ratio "B" is high, the design of the furnace is faulty, for other considerations may make such a ratio desirable or even necessary. The point that requires close attention is the most perfect heat insulation possible. If the heat insulation is good then, while there may be a great difference in temperature between the charge on the hearth of the furnace and the resistor, there will be very little difference between the rest of the surface receiving radiation and the resistor. Here was found the great weakness in Furnace 4, for the walls, instead of being good insulators, were relatively good conductors of heat. Observation showed that when a balance had been reached, so that the total energy radiated from the outside of the furnace was equal to the energy generated in the resistor, there was a marked difference between the temperature of the resistor and the interior surface of the furnace. There were no means by which these temperatures could be measured, but by the eye it could be seen that the difference amounted to at least 200° C.

A very clear demonstration, that was approximately quantitative, of the high heat conductivity of the walls was made by comparing Furnace 4A with Furnace 4. In Furnace 4A the side walls

were partly covered with bricks having high heat insulating properties, about 25 per cent. of the total exterior surface of the furnace being protected in this way. From the table it is seen that, working with even a higher temperature than in the case of Furnace 4, the losses were considerably reduced. It is unfortunate that the determinations were not made at the same temperature, but it is safe to assume that, if Furnace 4 had been tested at a temperature of 1200° C., the losses would have amounted to at least 43 kw. Hence by the improved heat insulation the loss was reduced about 19 percent.

In building Furnace 4, a good grade of fire brick, so far as its refractory properties are concerned, was used. It was recognized that silica brick are better heat insulators than fire brick, as may be seen from Wologdine's determinations,[2] which show that the heat conductivity of silica is 0.0020, and of fire brick 0.0042. On account of its high coefficient of expansion by heat, silica brick is sometimes objectionable, and it was thought that, with a wall of fire brick 460 mm. (18 in.) thick and an air gap, the heat insulation would be amply sufficient. The air gap, however, was useless, for a reason that has been well expressed by Ray and Kreisinger.[3] The surface of the inner wall forming the air gap reached a temperature of at least 600° C., so that the very device on which dependence was placed to prevent heat losses was worse than useless.

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Niagara Falls, N. Y.

DISCUSSION.

Mr. John Thomson (*Communicated*): I was much interested in Mr. FitzGerald's perspicuous note, and generally quite agree with the deductions and opinions expressed. There were, however, two internal conditions in the thermally inefficient furnace different from those previously tested, and it is believed, on

[2] "Electrochem. and Met. Industry," Vol. III (1905), page 291.

[3] "The Flow of Heat Through Furnace Walls," by Walter T. Ray and Henry Kreisinger; Bulletin 8, Bureau of Mines, Washington, 1911.

theoretical grounds, that they contributed somewhat to the excessive heat losses. These differences consisted, firstly, in the dimensions of the interlocked carbon resistor plates from which the dispersion of heat might take place quite as readily in the horizontal plane as in the vertical, instead of the reverse of this, as in the prior furnaces; and, secondly, that the outer vertical sides of the resistor were in considerably closer proximity to the brickwork than formerly. These conditions were not thought of in time to make any test as to their relative importance, hence, as mentioned, they are advanced upon a hypothetical basis.

That the "air-gap" is as useless as the quoted experiments by Ray and Kreisinger would make it appear is not, in the writer's opinion, completely proven. According to present recollection, Bulletin 8, of the Bureau of Mines, which reported their experiment, does not show that the temperature of the air within the gap was taken, nor that it was known whether the air was "dead" or displaceable by cold air from the exterior of the furnace. These conditions are obviously important, for if the heat radiated from the inner wall is at a sensibly higher temperature than the contained air, and especially if this air can be displaced by cooling air, then the precise formulated deductions are open to suspicion. While somewhat far afield, an illustration of the point involved may be found in cork—in fact, wood—which is an excellent insulator of heat and sound, and has been extensively used, under compression, as a powerful resistive spring or cushion. These advantageous features are due to the fact that cork is a cellular structure, each cell being complete and distinct from the others, consequently the imprisoned air or gas cannot escape. Now, to practically realize in a highly heated furnace a circumscribing gap which shall be but reasonably air-tight is, as anyone with experience will admit, not readily attainable. It is believed to be extremely doubtful if the gap was really an isolated space in either the furnace which Mr. FitzGerald and the writer designed or in the furnace referred to in the aforesaid Bulletin 8. If not, then the heat radiated from the inner surface of the gap-wall went more or less directly to the outer atmosphere. Moreover, the tendency to accomplish this inheres in one of the conditions produced by a rise of temperature, namely, the expansion, hence pressure, of the contained air. There are

plenty of good principles that have been damned because of ineffective construction of the means for utilizing them, but this particular one I do not think has been, however. Finally, the kilowatt difference, relative to the structural cost, as between an effective air-gap and the same space filled with brick-dust, or kieselguhr, or asbestos, is about as remunerative as the chase for the last place of decimals. The writer assumes his full share of responsibility for what would have been, at the best, a needless refinement, promises not to repeat the blunder, and would beg to be included with those who may offer thanks to Mr. FitzGerald for presenting the matter to the Society.

A paper read at the Twentieth General Meeting of the American Electrochemical Society, in Toronto, Canada, September 21, 1911, President W. R. Whitney in the Chair.

SMALL ELECTRIC FURNACE WITH HEATING ELEMENT OF DUCTILE TUNGSTEN OR DUCTILE MOLYBDENUM.

By R. Winne and C. Dantsizen.

There are many uses for a simple furnace which can be run higher than the melting-point of iron, and in which substances can be heated without taking up either carbon or oxygen.

There have been a few special furnaces, electrically heated, which are capable of fulfilling these conditions. Of these, we may mention the following:

1. The iridium-tube furnace. This is very fragile and extremely expensive.

2. A carbon-tube furnace with a porcelain tube inside. This makes an excellent tube-furnace, but calls for special transformers.

3. The platinum foil or wire-wound body of fire-clay or other refractory. This has been to the average man the most easily accessible and most generally used form of small furnace for fulfilling the conditions. But it has its limitations. Most serious of these are the fact that the platinum is easily contaminated, and that, except for small sizes, the winding is very expensive. Besides this, much of the work for which these furnaces are used necessitates running the platinum perilously close to its melting-point.

4. The Arsem vacuum furnace,* in which a graphite heater is used to generate the heat and in which the oxidation is prevented by the vacuum. This introduces carbon when very high temperatures are used.

We will show in the following how two simple types of furnaces have been made with a winding of ductile tungsten or ductile molybdenum. It is now possible to produce these substances in the form of wire or ribbon, and their high melting-points and relatively low cost adapt them admirably to the purpose.

*These Transactions, 9, 153-172 (1906).
J. Am. Chem. Soc., 28, 921-935.

Crucible Furnace.

This consists of a helix of ductile tungsten or molybdenum, supported by an alundum tube and protected from oxidation by a hydrogen atmosphere. The container, for the charge to be heated, is a small crucible which is placed inside of the alundum cylinder.

The diagram, Fig. 1, shows the furnace in vertical section. *W* is the heating wire, of ductile tungsten or molybdenum. It is wound on *G*, an alundum cylinder which is molded plain

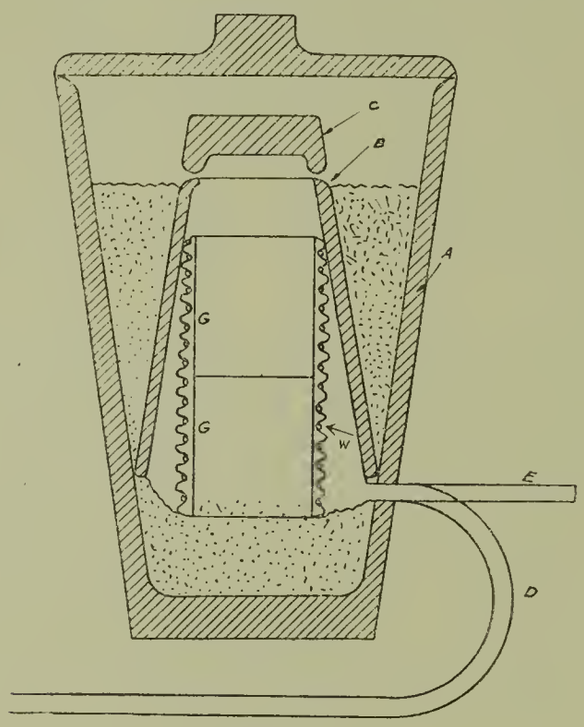

FIG. 1.

inside and with a helical groove on its outer surface. *A* is a Battersea crucible. *B* is an inverted Battersea of which the bottom has been cut off. *C* is the crucible bottom, used as a cover. The bottom of *A*, as well as the space between *A* and *B*, is filled with powdered silica. Hydrogen is constantly supplied to the furnace through the pipe *D*, and burns as it escapes from the top of *B*. This gives a protecting atmosphere for both the tungsten or molybdenum winding and the object to be heated. Current is supplied to the winding through two large copper connectors, *E*.

An object to be merely heated, as for annealing, may be inserted directly into the alundum cylinder.

Material to be melted is placed in a small crucible, and this

is let down into the alundum cylinder. A suitable crucible for holding the charge is made of pure alumina.

A few dimensions and details of construction may be of interest.

In the apparatus shown in Fig. 1, the Battersea crucibles *A* and *B* are sizes *O* and *J* respectively. The alundum body *G* consists of two cylinders placed end to end. Each is 7.6 cm. high and 5.1 cm. inside diameter, while the distance from one convolution of the helix to the next is .95 cm. These alundum cylinders may be obtained from The Norton Company, of Worcester, Mass. The winding consists of 260 cm. of square molybdenum wire, 1.27 mm. on a side. This wire is first wound on a mandrel 3.8 cm. in diameter, and wire and mandrel are then heated up to about 800° C. Upon then releasing the coil,

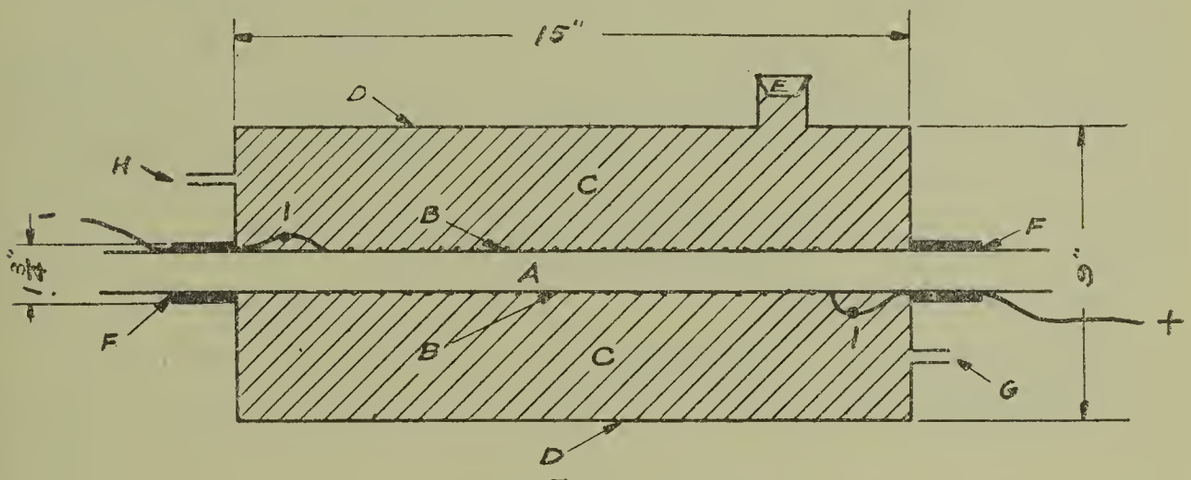

FIG. 2.

it expands to the diameter of the alundum body, on which it can then be screwed. The copper connectors, *E,E,* are 0.8 cm. in diameter, and the ends of the coil are simply clamped in with set-screws. The copper connectors and the hydrogen inlet tube are held in place in the crucible wall by a mixture of powdered silica and water glass, which at the same time prevents loss of hydrogen.

This furnace can be safely run up to 1700° C. At this temperature it calls for 25 volts and 45 amperes.

We have melted pure iron and made many iron and other alloys in this furnace.

Tube Furnace.

This consists of a porcelain or alundum tube, wound with tungsten or molybdenum foil. Around the tube is a tight metal

casing filled with powdered silica, and, to prevent oxidation of the winding, a hydrogen atmosphere is maintained in the casing.

Such a furnace is shown in the diagram, Fig. 2, in vertical length section. *A* is an alundum tube, 2.3 cm. inside diameter and 46 cm. long. The casing *D* is made of thin sheet iron and is oxy-acetylene welded. The winding is a molybdenum ribbon, 0.184 mm. thick, 2.54 mm. wide and 445 cm. long. *I,I* are heavy copper leads, fastened to the ends of the coil by twisting the latter about them. *C* is the powdered silica packing, *F,F* is some asbestos wool, used to prevent the escape of hydrogen at the ends of the casing. *H* and *G* are the hydrogen inlet and outlet tubes respectively. *E* is a rubber stopper.

With this furnace a temperature of 1600° C. is readily attained. At this temperature it calls for 80 volts and 14.3 amperes.

The two furnaces above described are adapted to heating the charge in an atmosphere of hydrogen. In case it were desirable to heat in an oxidizing atmosphere, it would be necessary to replace the very porous alundum by porcelain, as otherwise the furnace winding would become oxidized.

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DISCUSSION.

Mr. W. L. Morrison: How long does it take to heat up the furnace to 1700° C.?

Mr. C. Dantsizen: In order to preserve the economical life of the furnace, we take about an hour and a half to reach 1700°. We can, however, reach that temperature in half that time.

Dr. E. F. Northrup: I would like to ask if the authors have found any advantage in using silica instead of magnesia as their heat insulating material, and whether the silica, after a time, has any action whatever on the molybdenum to make it brittle or destroy the winding. Is molybdenum foil now obtainable on the market?

Mr. Dantsizen: I think for our purposes magnesia is just as good a heat insulator as silica. Silica does not affect the molybdenum at all, and does not therefore make it brittle.

Molybdenum can be obtained from the General Electric Company.

MR. CARL HERING: Can any other gas be used instead of hydrogen? I suppose illuminating gas would form carbides.

MR. DANTSIZEN: Illuminating gas could not be used because hydrocarbons do affect molybdenum and tungsten. Carbon monoxide might, however, be used, because it does not seem to form carbides with these metals. Carbon dioxide could not be used.

MR. L. E. SAUNDERS: In connection with the last statement in the paper, that if it was desired to heat in an oxidizing atmosphere, the alundum tube should be replaced by porcelain, I may say there will soon be supplied some alundum tubes which are made non-porous by means of a glaze on the inside.

A MEMBER: Anyone who has carried on experiments with alundum tubes knows that its porosity is a defect for certain experimental work. Could we get it dense enough so that it will not allow the permeation of gas?

MR. SAUNDERS: Yes. The melting point will still be high enough to stand 1700° C. It will be slightly lower than that of the original body.

DR. G. B. WATERHOUSE: In making this tube furnace, do the edges of the foil touch, or is there an appreciable space between them?

MR. DANTSIZEN: The metal is in direct contact with the alundum, and the turns are separated by a fraction of a centimeter.

MR. M. M. KOHN: Would the glaze which Mr. Saunders speaks of have any tendency to craze under high heat, or would it be a homogeneous structure?

MR. SAUNDERS: I have only seen three tubes glazed in this manner, and they were not "crazy."

DR. NORTHRUP: In accurate measurement work it is very necessary not only to get a high temperature, but also to get a temperature uniform over a considerable portion of the length of the furnace. Does the passing of a gas through the furnace tend to make one part of the tube cooler than another, or is it just as possible to get a uniform temperature in such a furnace as it is in the ordinary platinum-wound furnace?

MR. DANTSIZEN: It is just as possible to get uniform tempera-

tures in our furnaces as in a platinum-wound furnace, because the flow of the gas around the tube is very slow.

MR. KOHN: Is there not a possible danger of explosion of the hydrogen gas?

MR. DANTSIZEN: When we first built these furnaces, we feared explosion, but, strange as it may seem, we have had none.

MR. KOHN: Have you made any number of experiments?

MR. DANTSIZEN: Yes; the furnaces have been running every day for the last three months, and yet we have had no explosions.

PRESIDENT WHITNEY: Three or four furnaces running at a time, too, for several months. You have to be careful with the hydrogen; that is obvious.

MR. DANTSIZEN: Lack of explosions in these furnaces may be explained by the fact that the union of oxygen with hydrogen in the silica packing must of necessity be relatively slow. The openings of the furnaces are so large that even if an explosive mixture of hydrogen and oxygen should become ignited in the interior of one of them, the force of the explosion would not be sufficient to damage it.

MR. R. C. SNOWDON: Is it possible to coat a coil over the outside with some material like sodium silicate, or something of that character, to keep down oxidization, or does molybdenum-tungsten take the oxygen away and leave the sodium free, or something of that kind?

MR. DANTSIZEN: It is quite possible to protect the molybdenum-tungsten with some substance, such as an oxide, but sodium silicate cannot be used, because that reacts with molybdenum and tungsten. Up to a certain temperature it can be wound and packed with cement.

PRESIDENT WHITNEY: About how high do you estimate you can run the temperature and not affect the cement which the Norton Company sell with the alundum?

MR. DANTSIZEN: Their cements hold up as well as the alundum does itself.

MR. SNOWDON: I had in mind a temperature somewhere about 1800° C.

PRESIDENT WHITNEY: You mean, to protect the wire from oxidation by the use of cement?

MR. SNOWDON: Yes.

PRESIDENT WHITNEY: The ordinary cements do not do that.

A paper presented in abstract by Mr. Carl Hering, at the Twentieth General Meeting of the American Electrochemical Society, in Toronto, Canada, September 21, 1911. President W. R. Whitney in the Chair.

DESIGN OF A 30-TON INDUCTION ELECTRIC FURNACE.

By Albert Hiorth.

The design of this furnace has been based upon the data and results obtained with the original 5-ton furnace in Jossingfjord, Norway, as shown in the daily report sheets.

As the step from 5 tons to 30 tons is a rather large one, it was necessary to carefully study beforehand the different factors determining the design. The two most important things to be determined were the power-factor, and the energy required to keep the charge at the working temperature.

The power-factor depends almost exclusively upon the design of the furnace and the periodicity of the supply. With a given resistance in the bath, a certain current is necessary to produce the required temperature. As the bath consists of only one turn round the magnet, the secondary current is equal to the primary current multiplied by the number of primary turns per leg.

The voltage required to pass this requisite current through the primary winding may be divided into two component parts, *viz.*, one required to overcome the ohmic resistance ("The Resistance"), and the other required to overcome the inductive resistance ("The Inductance").

These two factors determine the power-factor of the furnace. With a given inductance the power-factor increases together with the resistance, and with a given resistance the power-factor decreases as the inductance increases.

The total resistance may again be divided into three parts: First, the primary resistance which represents the loss in the primary coils; second, a very small part representing the loss in the iron-core; third, the secondary resistance representing the resistance of the bath, and which may be termed the useful resistance.

This useful *resistance* depends upon the following three factors: The conductivity of the iron at the working temperature of the furnace; the skin effect; the dimensions of the bath. The first of these factors will be dealt with later; the second, the skin effect, can be neglected for our purpose, as it may safely be

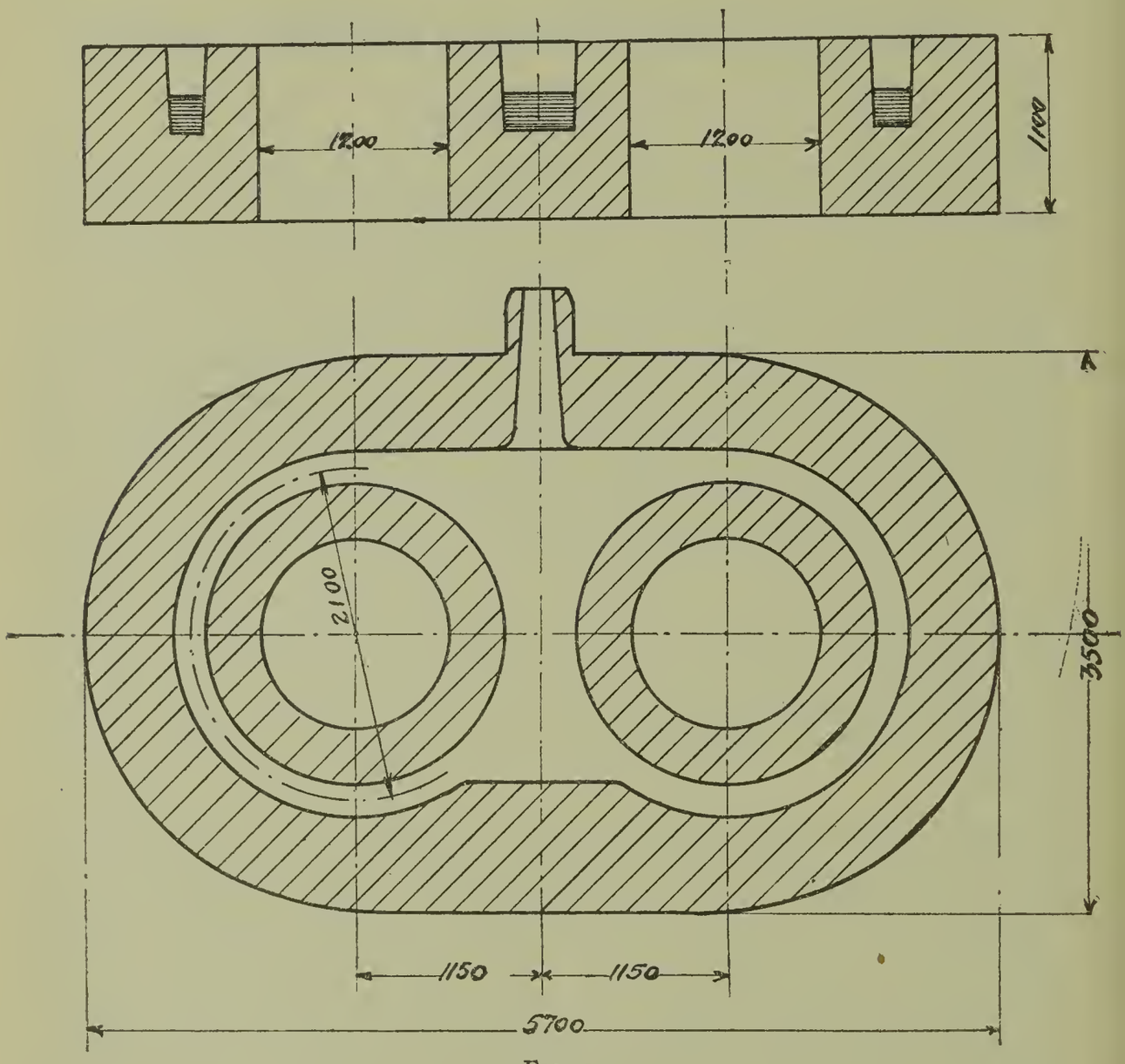


FIG. 1.

assumed that its effect upon the total resistance will be the same, even if the sectional area of the bath is altered considerably.

The third factor, the dimensions of the bath, is, of course, the main one. If the skin effect is neglected, the resistance increases proportionally with the length of the bath, and decreases proportionally with the sectional area.

With a given periodicity the *inductance* depends upon two factors: First, the magnetic resistance, *viz.*, the "reluctance" of

the iron-core; second, the magnetic leakage between the primary windings and the bath.

The first of these may be neglected, as the magnetising current of the furnace without any charge is only very small.

The second one, the magnetic leakage, depends upon the total flux which passes round any one of the windings (the primary winding and the bath) without also passing round the other; in other words, upon the total flux which escapes through the annular spaces between the two windings.

If the radial width of both the bath and the primary coils remains the same, this flux is practically proportional to the total area of the space between the winding and the bath, and if the distance between these two be not altered, the flux is proportional to the circumference of the bath.

Consequently the inductance will increase with the diameter of the coils; if at the same time the radial width of the bath be increased, the inductance will be somewhat reduced again, but not nearly proportional to the increase in the width.

The inductance is also, of course, proportional to the periodicity, and may be lowered by using a lower periodicity. The influence of this will be more fully discussed later on.

The design of the present furnace is shown in Fig. 1. The data for the furnace are the following:

Mean diameter of the annular space occupied by the bath, 2.1 meters.

Width of this space, 20 cm.

Depth of the bath with a 5-ton charge, 27 cm. (specific gravity of the iron assumed to be 7).

The primary coils have 15 turns on each leg, 8 below and 7 above the bath. Each turn has a sectional area of approximately 1,000 sq. mm. The resistance of each leg at a temperature of approximately 180° C. is 0.003 ohm.

The weight of copper per leg, 840 kg.

The core is approximately 40 by 50 cm., giving an effective sectional area of 1,800 sq. cm.

The weight of the core is approximately 15 tons. The periodicity of the supply is 12.5 complete periods, corresponding to 25 alternations per second.

From the daily report sheets recording volts, amperes and

kilowatts, taken at intervals of about half an hour and extending from the beginning of July until the middle of October, 1910, we have selected six at random, and from these plotted diagrams showing the readings as ordinates, with the time as abscissæ Three of these diagrams are shown in Figs. 2-4. These are for the charges No. 45 (which was the charge witnessed by Professor Jos. W. Richards[1]), No. 50 and No. 63. During the two first ones, pig iron was melted and converted into steel, whereas the last one was run for the purpose of refining low-grade steel.

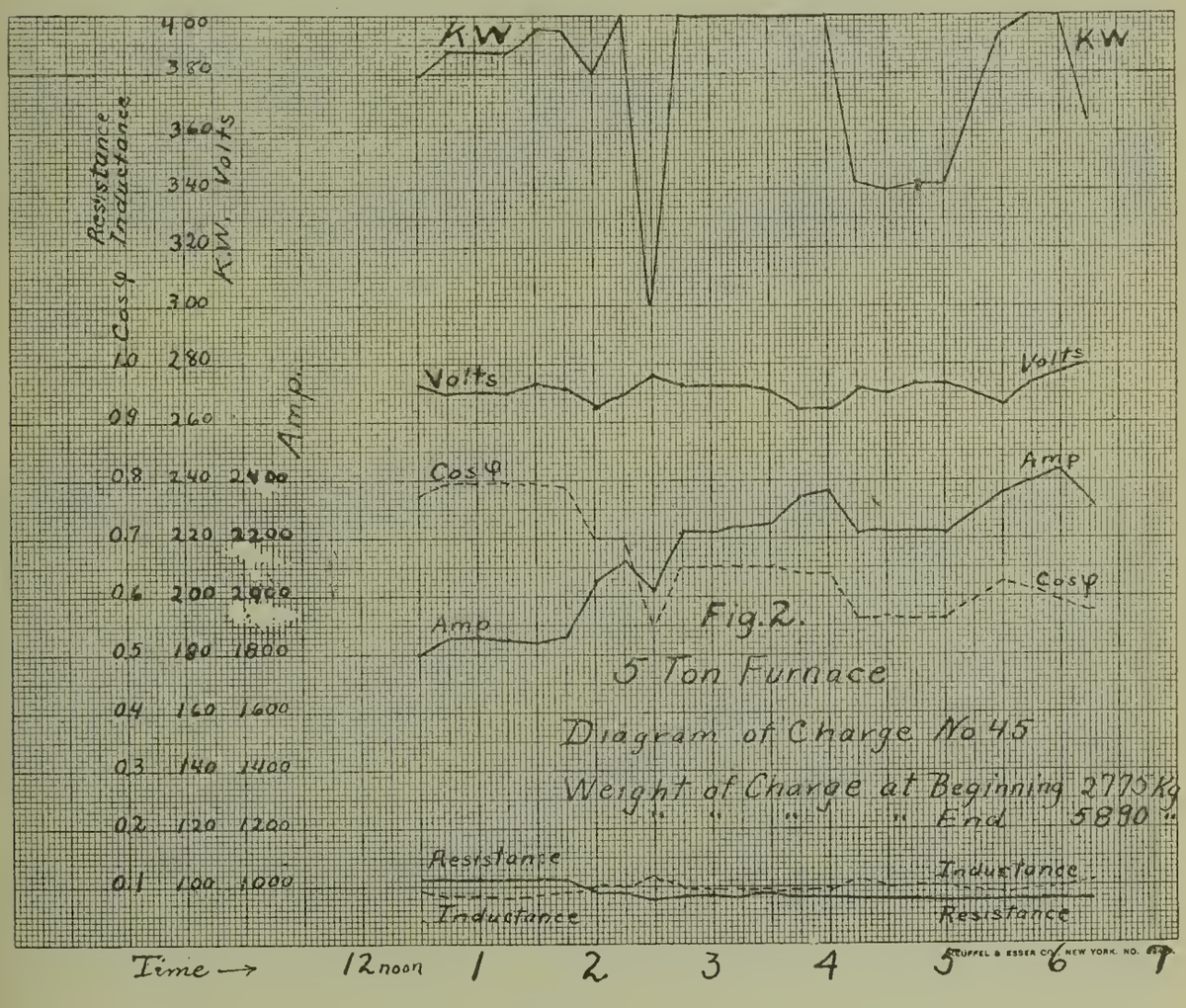
In scrutinizing these curves, it will be found that the electrical conditions have varied considerably, and, in fact, more so than one is accustomed to expect in an induction furnace. This is partly due to the fact that the curves are plotted from actual readings on indicating instruments instead of being traced by recording instruments. In this way the peaks in the curves are naturally more pointed than would otherwise have been the case, and partly caused by the voltage of the generator being varied a good deal in order to alter the temperature of the bath.

Some of the peaks are evidently caused by the lifting of the upper primary coils in order to gain access to the bath, either for inspecting purposes or in order to work the slag or to add material to the bath. An example of this last named disturbance is shown very clearly at 2.30 P. M. during charge No. 45. In this case the voltage has been kept constant and the inductance has been suddenly increased, due to the lifting of the upper coils. This causes a drop both in the amperage, the power-factor and the energy, although the voltage was not altered. These diagrams also show the variations in the power-factor, the resistance and the inductance. These last named have been arrived at by splitting the voltage up into its two component parts, as explained on page 1, and dividing these by the current. The results thus obtained represent directly the resistance and the inductance, as defined on page 294.

As has already been explained, the resistance and (with a given periodicity) the inductance are constants, which depend upon the dimensions of the bath, the weight of the charge (which depends upon the sectional area of the bath) and upon the disposition of the primary coils relative to the bath.

[1] These Transactions, **18**, 200, (1910).

Considering the resistance first, this should, in accordance with the above theory, remain the same as long as the weight of the charge is the same. Apart from the variations caused by the adding of fresh material to the bath, there is, however, a marked decrease of the resistance as the process progresses; whether this is due to the alterations in the chemical composition, or whether the conductivity (see page 294), after a certain tem-



perature has been reached, begins to increase again with a further increase of temperature, cannot be stated definitely without further investigations.

This decrease of the resistance in the course of the run is shown very clearly on the diagram, Fig. 2, and also toward the end of the diagram, Fig. 3. In Fig. 4 this phenomenon is not observed, perhaps because this charge was only for refining purposes. It appears from the diagram as if the resistance was somewhat higher during the night, when the temperature was

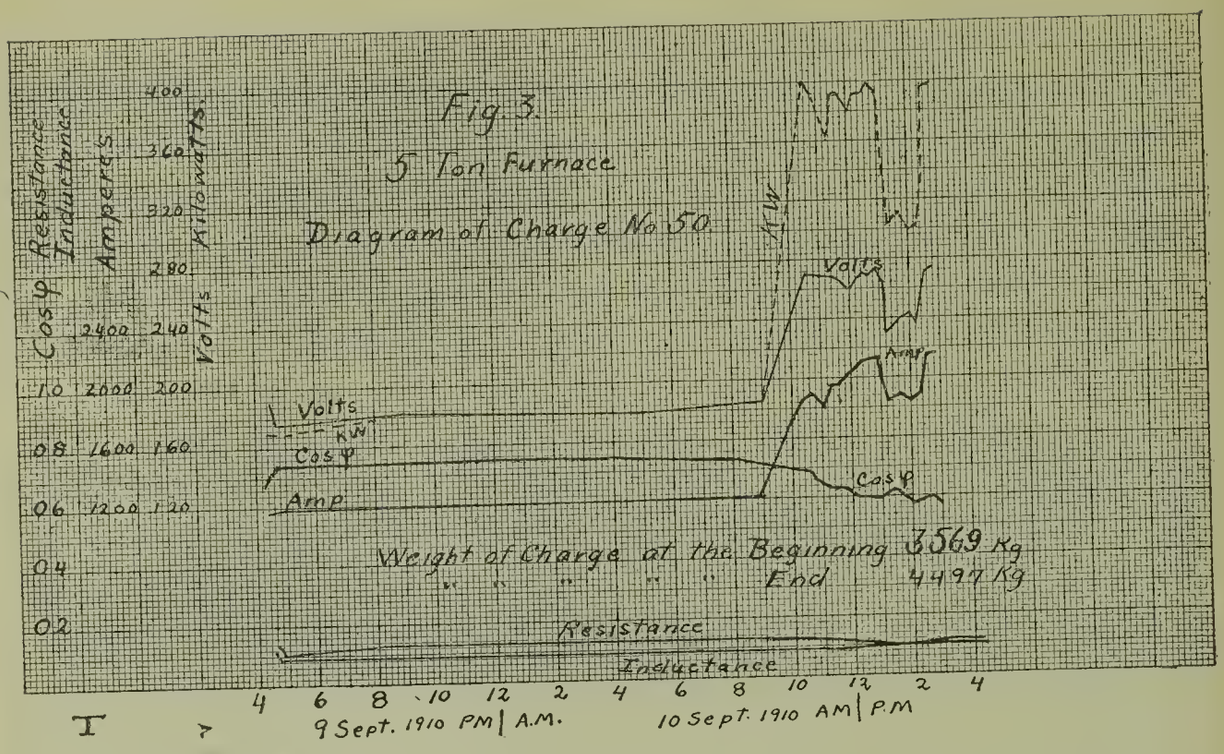
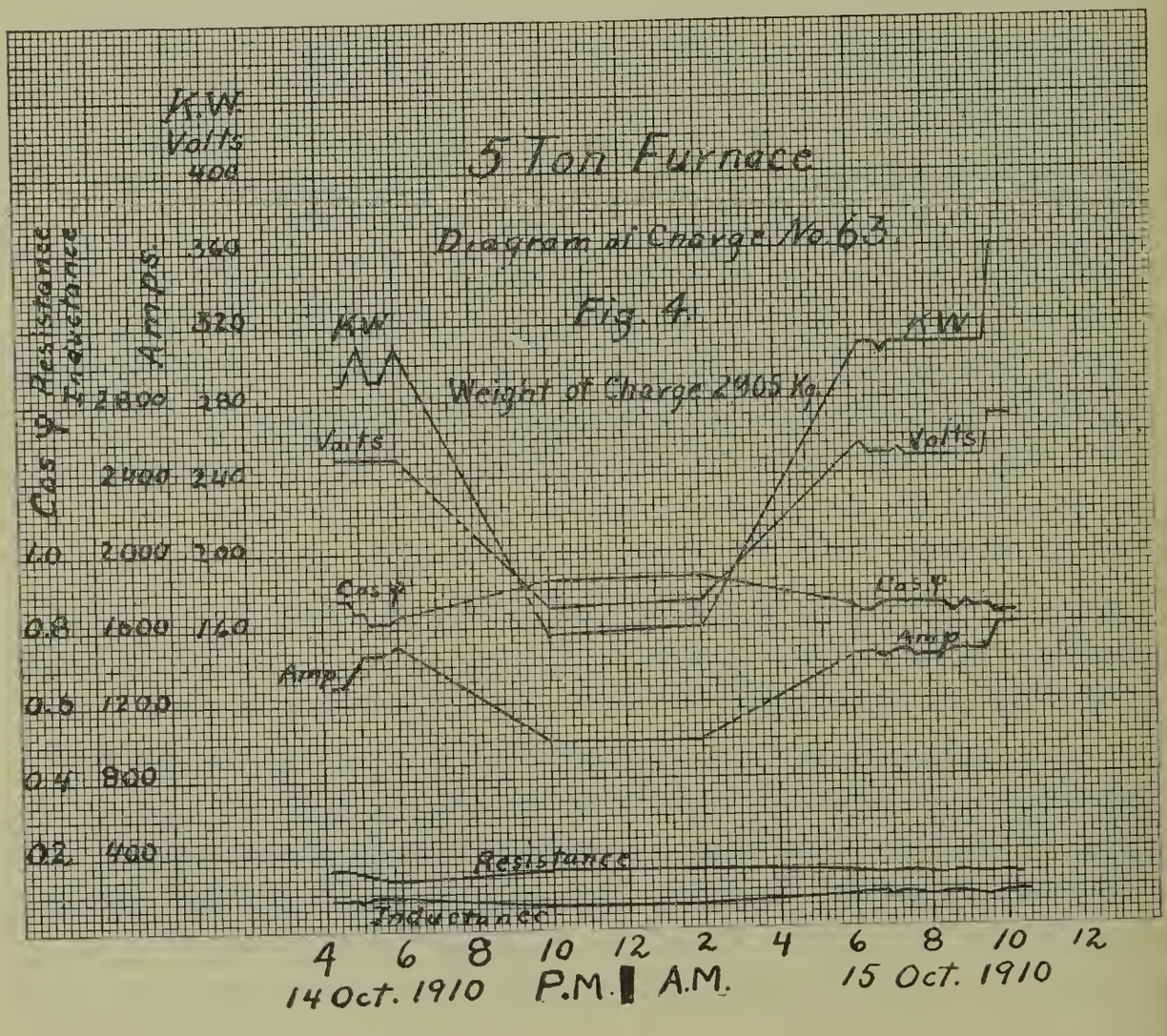


Fig. 3.
5 Ton Furnace
Diagram of Charge No 50
Cos φ Resistance Inductance
Amperes
Volts
KW
Volts
Amp
Cos φ
Weight of Charge at the Beginning 3569 Kg
" " " " " End 4497 Kg
Resistance
Inductance
9 Sept. 1910 PM | A.M.
10 Sept. 1910 AM | P.M


K.W.
Volts
5 Ton Furnace
Diagram of Charge No 63.
Fig. 4.
Cos φ Resistance Inductance
Amperes
KW
Weight of Charge 2905 Kg.
Volts
Cos φ
Amp
Resistance
Inductance
14 Oct. 1910 P.M. | A.M. 15 Oct. 1910

lower, but as the inductance at the same time appears abnormally low, it is safer to assume that the readings, being at the lower part of the scale, are somewhat inaccurate.

If we consider only the resistance at the end of a run or at the beginning, before fresh material is added, we obtain the following figures for the resistance from the reports analyzed:

Charge No.	Weight kg.	Sectional Area sq. cm.	Total Resistance Ohms	Equivalent Useful Resistance Ohms
8	1475	160	0.390	0.380
"	5032	545	0.094	0.086
29	2532	273	0.128	0.120
"	5878	635	0.066	0.058
41	3600	390	0.124	0.116
"	5764	623	0.070	0.062
45	2775	300	0.125	0.117
"	5890	630	0.070	0.062
50	3569	386	0.125	0.117
"	4497	485	0.088	0.080
63	2905	314	0.140	0.132

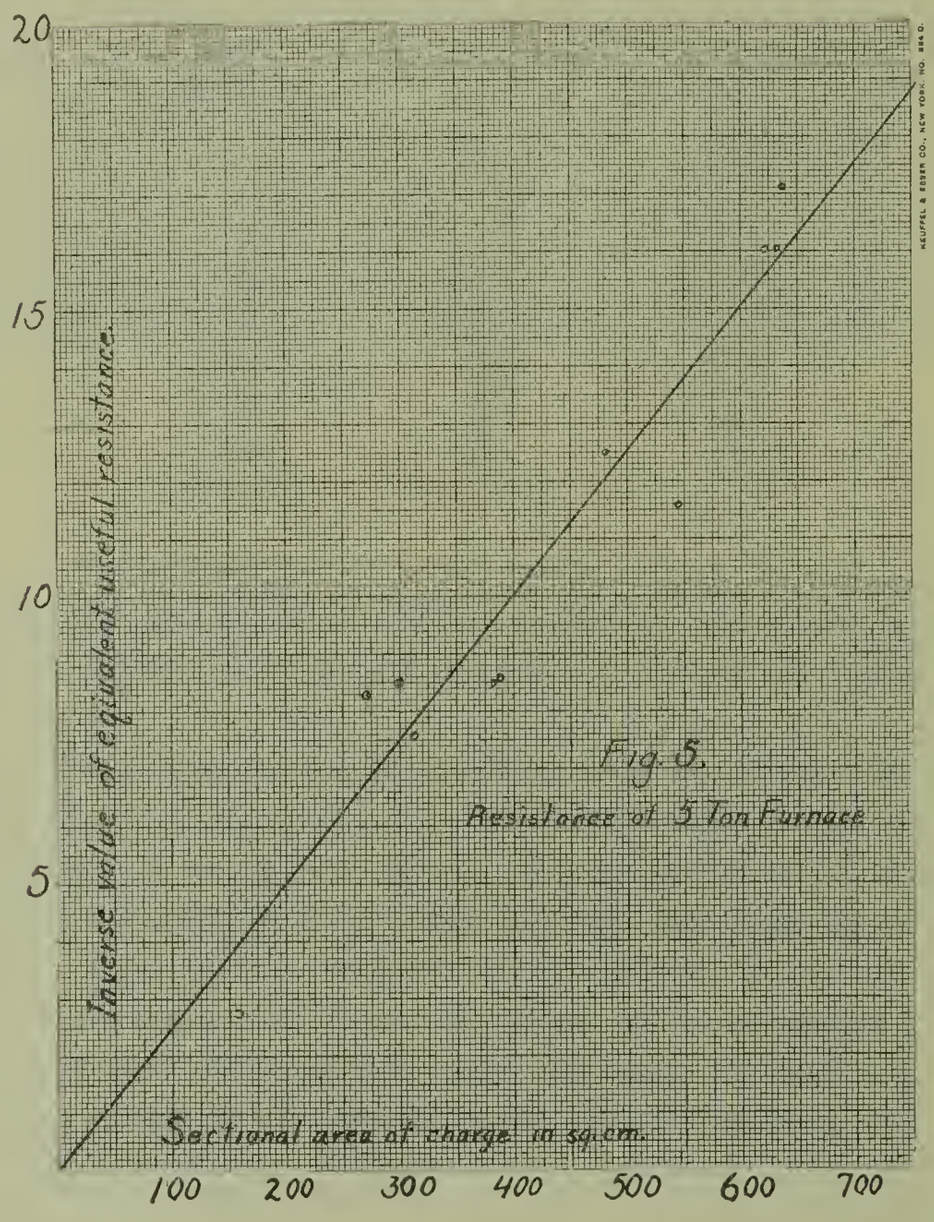
The last column in this table gives the equivalent useful resistance, *viz.*, the total resistance of the bath (twice the resistance of one ring) multiplied by the square of the number of primary turns.

In order to obtain this equivalent useful resistance, the total resistance has to be reduced by an amount corresponding to the resistance of the primary coils and to the losses in the iron-core. As stated on page 295, the resistance of the primary coils is 0.003 per leg = 0.006 for the whole furnace. The iron losses may be taken to represent the resistance equal to 0.002, corresponding to an iron loss of approximately 1-2 percent, and the total of these will thus be 0.008. Subtracting these figures from the total resistance, we obtain the equivalent useful resistance given in the last column.

As the resistance varies in inverse proportion to the sectional area, the curve showing the relation between the sectional area and the inverse value of the resistance should be a straight line. In the diagram, Fig. 5, the points obtained are marked as dots, and the straight line drawn so as to represent the average of the readings.

In figuring the actual resistance from this curve, one obtains a specific resistance of the molten iron equal to 1.4 ohms per

sq. mm. cross-section and m. length. In handbooks the value given is somewhat higher, and even as high as 1.7. The discrepancy may be due to inaccuracies, which are quite unavoidable, considering the difficult nature of the measurements, or may be due to the fact that in our case the section of the bath



is not absolutely uniform and that, for instance, the central portion of the bath, where the two rings meet, represents a somewhat lower equivalent resistance than the rest. The value 1.40, it must be remembered, includes all the irregularities in the shape of the bath, and also the skin effect, as far as this influences the resistance. It can only be used for figuring the resistance of furnaces of similar shape and disposition of the bath.

As mentioned on page 294, the inductance is independent of the weight of the charge, and remains constant at all times. It will be seen from the diagrams presented that the inductance varies between 0.09 and 0.10, except when the upper coil is lifted, and during the night (as in case of charge No. 63); but as the readings in this case, as already pointed out, are probably inaccurate, we may neglect this part of the diagram. All the other diagrams correspond to those shown, and the value of the inductance for the old furnace may therefore be taken as 0.095.

Using the figures thus established for the resistance and the inductance, we can figure out theoretically the power-factor of the present furnace with different charges, and obtain the following table:

With	3	tons	Cos. φ	= 0.81.
"	4	"	"	= 0.73.
"	5	"	"	= 0.65.
"	6	"	"	= 0.60.

As will be seen, this corresponds very well with the values given on the curves representing the different charges.

We are now in possession of the necessary figures to proceed with the design of the large furnace and to base it upon the experience with the old one.

With regard to this design, the first condition, of course, is that the space for the charge must be large enough to hold 30 tons, but the actual dimensions and shape of the bath must be such as to give the best possible power-factor.

In order to increase the capacity of the furnace, it is necessary to increase the sectional area of the bath more than the length, as otherwise the diameter of the bath, and consequently the dimensions of the furnace, would become too large. The resistance of the bath will thus be considerably decreased, and at the same time the inductance will be increased, owing to the larger diameter, which increases the area of the space between the primary and the secondary windings.

The power-factor will thus in any case be reduced with the increased capacity, and we have figured that a 30-ton furnace built on the same line as the present one, for single-phase current, with a two-leg magnet and a current supply with 25 alternations,

would show a power-factor equal to 0.25, and that the power-factor even with 15 alternations per second would still be only 0.38.

It was therefore decided by me that the 30-ton furnace should be built on the three-phase principle instead of the single-phase. In this case the weight of the charge per ring is only 10 tons instead of 15 tons with a single-phase furnace, and, as will be shown later, the reduction of the power-factor is not nearly so great.

In order to facilitate the tilting of the furnace, the three rings were disposed in one row. This arrangement is not strictly symmetrical, and it is to be expected that the load on the middle phase will be somewhat different from the load on the two outer ones, on account of the difference both in the inductance and in the resistance. With a special supply this is, however, not a very serious drawback, particularly as the difference between the phases in all probability will not be very great.

The dimensions finally chosen for the new furnace are shown in Fig. 6; from this we may now determine the resistance and the inductance in order to arrive at the power-factor.

Using the figure 1.40, the useful resistance figures out to be approximately 0.0001.

In order to arrive at the total resistance, this has to be increased approximately 7-10 percent on account of the losses in the primary winding and the iron-core, and the total equivalent resistance based on one winding becomes then 0.00011.

The inductance of the old furnace was 0.095, or, reduced, to one turn, 0.00021. In the new furnace this will be increased in proportion to the diameter, that is $\frac{0.00021 \times 3}{2.1} = 0.0003$.

These values of the resistance and the inductance would give a power-factor equal to 0.34, which is still too low.

In order to increase the power-factor, it is necessary to further decrease the inductance by using a lower periodicity. What can be obtained in this way is shown in the following table:

Periodicity	Power-factor
25 alts.	0.34
20 "	0.41
16 "	0.50
10 "	0.67

From this table we select a periodicity of 16 alternations per second. To decrease the periodicity further still will hardly pay, seeing that the cost of the generator will thus be further increased, even if the output is reduced on account of the power factor being improved. This, however, is a matter which should be gone into more closely later on, in connection with the actual cost of the generator.

It remains to figure out the necessary energy required for the furnace, the dimensions of the iron-core and the number of primary turns, together with the sectional area of these.

As the furnace will be used with a liquid charge, and no melting-down will take place, the amount of energy required is only the amount necessary to keep the bath at the required temperature. Apart from the small losses in the primary coil and the iron-core, all the energy is converted into heat in the charge itself, and, as soon as a constant temperature is reached, finally conducted to the surface of the furnace and given out from this by radiation, conduction and through the cooling arrangements. If the water-cooling is arranged in the same way and the amount of water increased correspondingly, it may therefore be assumed that the loss of heat, and consequently the energy required to keep the charge hot, will be in proportion to the surface of the furnace.

From Fig. 6 we determine the surfaces of the old and new furnace, as follows:

		New Furnace	Old Furnace
Inside circular surface	(*A.*)	20.5 sq. m.	8 sq. m.
Outside surface	(*B.*)	35. "	17 "
Upper "	(*C.*)	28. "	15 "
Lower "	(*D.*)	28. "	15 "

The surface of the new furnace is consequently between two and two and a half times as large as the old one. The amount of energy required in the old one to keep the charge in a molten condition during the night was approximately 180 kw., and during the refining the energy varied between 250 kw. and 350 kw. The minimum energy required for the new furnace should consequently be $2 \times 180 = 360$, and the maximum, $2.5 \times 350 = 875$. We have assumed 700 kw., but at the same time the generator should be constructed in such a way that, by merely

altering the voltage, it can give anything between 400 and 1,000 kw. With 700 kw. the current per ring becomes approximately 46,000 amperes, and the current density in the bath will be about 0.34 ampere per sq. mm.

The further data of the furnace, based upon the figures arrived at above, are:

Iron core, 1,200 sq. cm. Weight, about 23 tons.

Primary windings; voltage, 230 volts.

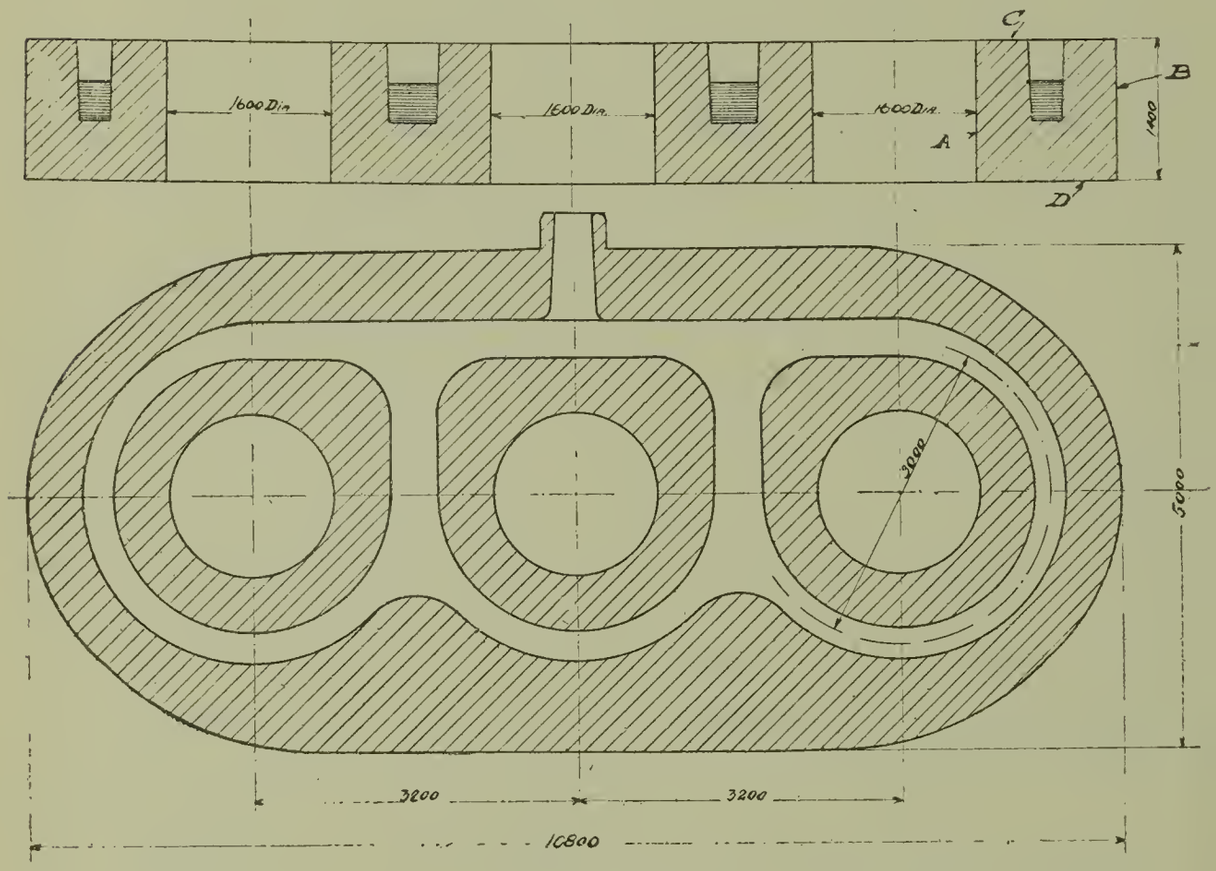


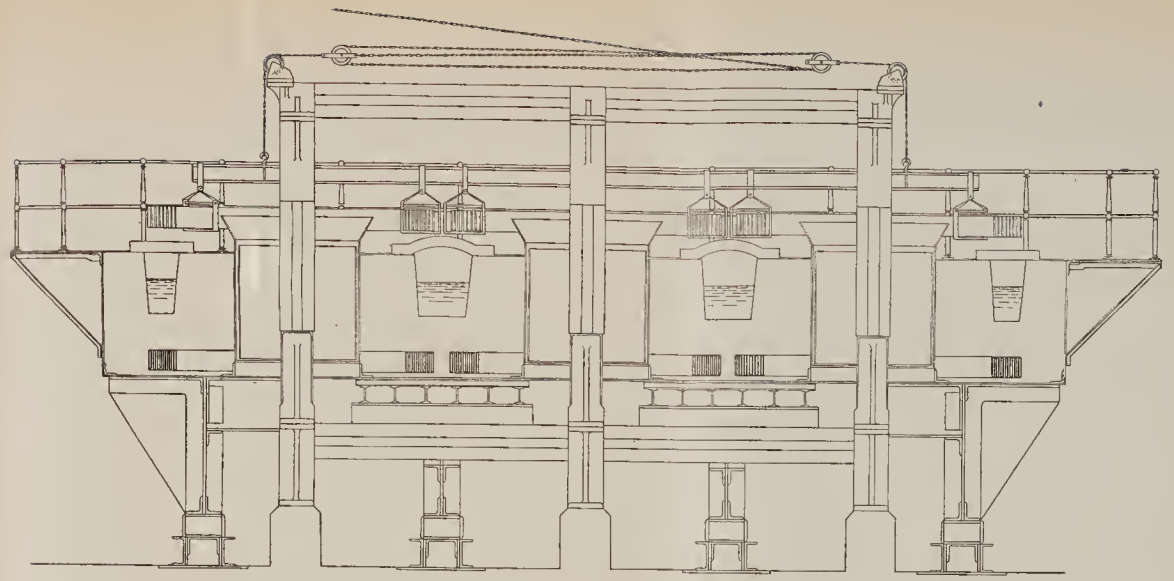
Fig. 6.

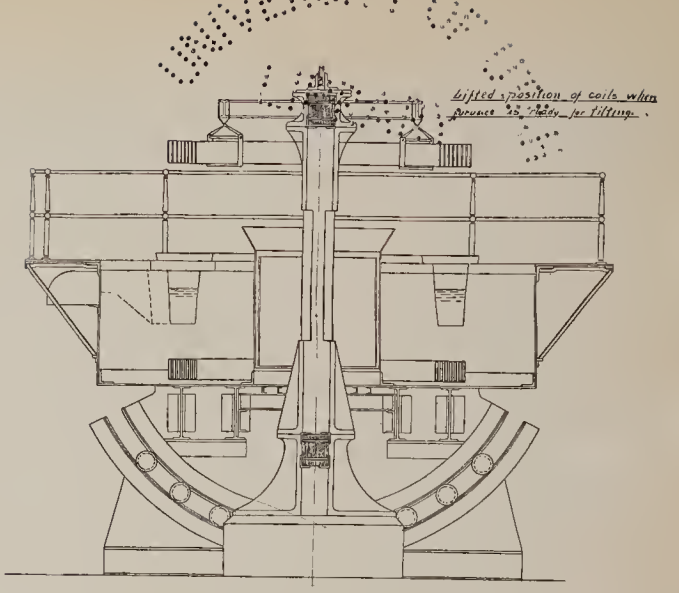
Number of turns per leg, 13, disposed 7 below the bath and 6 above.

Sectional area, 2 x 20 cm.

Weight of copper for the whole furnace, about 13.5 tons.

As stated above, the theoretical number of turns is only 13, but in order to be able to run the furnace at full charge, even if the inductance and resistance should prove to be somewhat different from the values given, two turns should be added per leg, making the number of turns in the upper coils 8. The last three turns in each of these coils should be provided with terminals, enabling one to use at will 12, 13, 14 or 15 turns per leg.





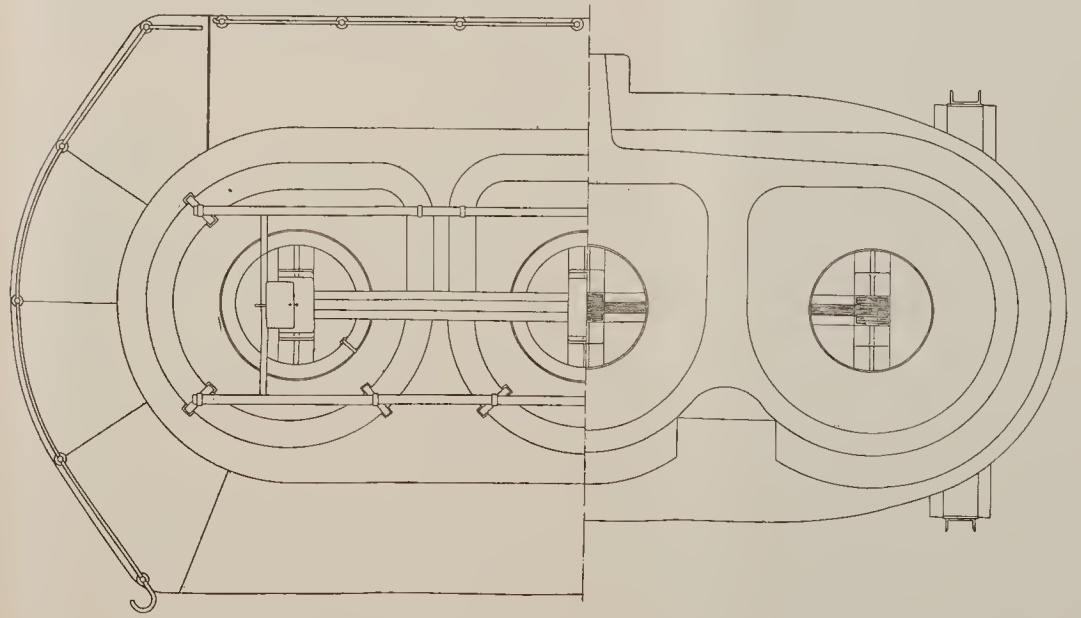


Fig. 7.

General Arrangement of

30 Tons Furnace.

Albert Sionth's Patent.

M = 1:75.

In Fig. 7 is shown a general arrangement of this furnace, designed on the same lines as the old one. The lower coil is embedded in the masonry below the bath, and consists of copper tubes arranged for water-cooling. The upper coil may be lifted in order to inspect the bath.

The inner surface of the furnace, marked *"A"* on Fig. 6, is also arranged for water-cooling.

In order to keep the covers for the bath small, the channels were given the form shown in Figs. 6 and 7. The primary coils have been shaped accordingly to follow the shape of the bath as closely as possible.

The primary voltage has been fixed at about 230 volts, as this low voltage greatly facilitates the construction and the insulation of the windings. To embed the coil in the masonry of the furnace would, for instance, be impossible with a higher voltage, and also the upper coil would have to be built and cooled in quite a different manner if it had to carry high-tension current. The use of high-tension current would therefore necessitate a considerably increased distance between the bath and the coils, and thus a considerably lower power-factor.

The table below shows the principal data for the old furnace and the new one:

	Old Furnace	New Furnace
Total capacity	5 tons.	30 tons.
Capacity per leg	2.5 tons.	10 tons.
Diameter of bath	2.1 m.	3 m.
Width of bath	20 cm.	30 cm.
Depth of bath	27 cm.	45 cm.
Total surface of masonry	55 sq. m.	110 sq.m.
Total length of platform	8¼ m.	13 m.
Total width of platform	5¼ m.	6.5 m.
Sectional area of core per leg	1800 sq. cm.	1200 sq. cm.
Weight of core	15 tons.	23 tons.
Number of primary turns per leg	15	13 (see p. 12).
Sectional area of primary windings	1000 sq. mm.	4000 sq. mm.
Weight of copper per leg	0.875 tons.	*ca.* 4.5 tons.
Total weight of copper	1.75 tons.	*ca.* 13.5 tons.
Energy used	250 kw.	700 kw.
Copper losses	12.3 kw.	40 kw.
Iron losses	18 kw.	18 kw.
Power Factor	0.65	0.50
Voltage	250	230
Periodicity (½ alternations per second)	12½	8
Amperes per phase	1400	3540
Kind of current	1 phase.	3 phase.

Table showing Electrical Conditions of 30 Tons Furnace
with varying Resistance and Inductance

Fig. 8.

No	Variations in		1 Min' 2. Norm 3. Max				Cosf	Remarks.
	Resistance	Inductance	№	K.W	Volts	Amp.		
1	±0	±0	1	408	175	2700	0,5	
			2	700	228	3540		
			3	1000	273	4240		
2	+25%	±0	1	432	175	2540	0,58	The charge may be increased to 37,5 Tons without reducing the powerfactor more than to.5. The max and min energy as well as current and voltage will then be the same as in the first case
			2	700	220	3180		
			3	1000	263	3800		
3	-25%	±0	1	349	175	2885	0,40	If the charge is reduced to 22,5 tons, the powerfactor will be .5 and the conditions again the same as in the first case.
			2	700	248	4090		
			3	775	261	4300		
4	±0	+25%	1	288	175	2275	0,42	If the number of turns is reduced to 12 and the charge at the same time reduced to 24 Tons, the powerfactor will be .5, and the max Energy 955 K.W. at 275 Volts 4000 Amp.
			2	700	273	3540		
			3	710	275	3570		
5	+25%	+25%	1	329	175	2170	0,5	By reducing the number of primary turns to 12, the max. Energy may be increased to 955 at 275 Volts, 4000 Amp Powerfactor = .5
			2	700	255	3160		
			3	815	275	3420		
6	-25%	+25%	1	234	175	2370	0,325	If the charge is reduced to 18 tons and the number of turns at the same time reduced to 12, the max Energy will be 955 K.W. at 275 Volts, 4000 Amp. Powerfactor = .5
			2					
			3	578	275	3725		
7	±0	-25%	1	611	175	3310	0,61	By increasing the number of turns to 15, the charge may be increased to 40 tons The electrical conditions will then be as in the first case
			2	700	187	3540		
			3	1000	224	4240		
8	+25	-25%	1	628	175	3000	0,69	By increasing the number of turns to 15, the charge may be increased to 50 Tons The electrical condition will then be as in the first case
			2	700	184	3160		
			3	1000	221	3800		
9.	-25	-25	1	550	175	3630	0,5	By increasing the number of turns to 15, the max. Energy will be 1000 K.W and the conditions otherwise the same as in the first case
			2	700	197	4090		
			3	770	207	4300		

The generator should, according to the foregoing, be designed for a normal output of 700 kw., 8 periods (corresponding to 16 alternations) per second, 230 volts, 3,540 amperes, and a power-factor of 0.5. At the same time it must be able to give 400 kw. at 165 volts, and 1,000 kw. at 275 volts, with the same power-factor, and 290 and 125 kw., respectively, with a power-factor of 0.362. The maximum current will thus be 4,300 amperes.

The engine or turbine driving this generator should be for a maximum output of 1,550 effective h. p., and run at a practically constant speed at all loads between 600 h. p. and 1,550 h. p.

This plant should be quite sufficient to supply energy to the 30-ton furnace, even if the constants of this should vary considerably from the figures given above. In order to study this, we have prepared the table, Fig. 8, showing what would happen, and the maximum and minimum energy which could be supplied to the furnace, in case either the resistance or the inductance or both should vary 25 percent from the assumed value, either up or down.

This table shows the reason why the upper coils should be provided with connections, such as described on page 304, as these coils enable one to compensate for even quite considerable variations both in the resistance and the inductance.

Kristiania, Norway,
March 27, 1911.

DISCUSSION.

Mr. A. H. Strong (*Communicated*): Mr. Hiorth's paper is very interesting, but there are one or two points which our engineers have brought up in connection with Mr. Hiorth's new design of induction furnace which I think might be mentioned.

The disadvantage of lifting induction coils to rabble the slag would seem rather serious, unless this operation can be done with little loss of time, as these furnaces would probably be

used chiefly for refining hot metal where two or three slags is a common thing.

In an induction furnace the magnetic leakage can be lessened, and therefore the induction, thus increasing the power factor, as in the Roechling-Rodenhauser type of induction and resistance furnace, by winding an auxiliary secondary coil directly over the primary, and leading this current to steel pole-plates set beneath the lining, which plates act as resistors of the second class (these Transactions, **15**, 173, 1909) ; this secondary current, fed to the resistance plates, and then led to the bath, increases the power factor from 0.5 to 0.8 in an 8-ton furnace. These pole-plates usually convey 20 to 30 percent of the total energy. Another method of increasing the power factor for any induction furnace is to connect an overexcited synchronous motor on the same circuit with the furnace, and beyond it on the same line. Many motors for constant service in steel mills, that is, running air compressors, etc., can be of the synchronous type. They have been so made that they correct the power factor of a large mill to a degree out of all proportion to their horse-power rating The cost of constructing these overexcited synchronous motors (also called synchronous rotary condensers) is by no means prohibitive in first cost, nor in operation ; in fact, it has been shown in some large plants that this is the best way to correct the power factor which is reduced by a heavy inductive motor load. With these two aids for increasing the power factor of induction furnaces, 25 cycles can be used in furnaces of 30 or 50-ton size, and still obtain a power factor as high as 0.7 or 0.8.

The low voltage to which the radial induction coils of Mr. Hiorth's furnace limits itself is not prevalent in the Roechling and Rodenhauser type, as there the primary coil is wound directly around the iron core as in transformers, and higher voltages can be used, such as 2,200 or even 5,000 volts, which is the voltage used on one of the 8-ton furnaces which has been in operation for four years.

One of the remarkable features with this 30-ton Hiorth furnace is the low effective power provided for, namely, 1,000 kw. This would limit the furnace to hot charges only. This furnace would not have a 30-ton output if cold stock were also to be treated. This 1,000 kw. for a 30-ton induction furnace compares most

favorably with the 15-ton Héroult furnace, which consumes about 2,000 kw. for refining molten metal. Experience would, no doubt, show whether it would be advisable to increase this 1,000 kw. for the 30-ton Hiorth furnace to refine molten metal more rapidly.

With a constant inductance (since the power factor increases with the increasing resistance) it is well to lengthen the bath, keeping the cross-section the same, thus increasing the resistance. The usual oblong form wound transformer coil lends itself somewhat better to accomplish this than a radial coil, with its proportionally larger circumference.

Mr. Albert Hiorth (*Communicated*): With regard to Mr. Strong's remark about the lifting of the coils, I beg to say that this operation by a practical construction and balance is so easy that the coils are lifted in a few seconds by a single handle. The construction of this furnace was undertaken especially for steel men, who desired to get a furnace to keep the steel hot some time after it has been tapped from the Bessemer convertor or open-hearth furnace.

Mr. Strong further remarks that the magnetic leakage could be lessened by using the Roechling-Rodenhauser arrangement of a secondary winding ending in pole-plates in the masonry. I beg to point out that I, before the construction of Roechling's furnace, had put up such extra secondaries ending in electrodes in the slag. In this respect I refer to my German patent, No. 216,734. Such an auxiliary secondary winding complicates to a great extent the construction of the furnace, which was designed to secure the simplest construction and working, as well as cheapness. In reply to Mr. Strong's criticism of the primary coils, and the low voltage in my furnace, I regard this to be a great advantage, as, in the first instance, every danger for the laborers is avoided, and, secondly, the coils are stiff and easy to handle, and no insulation or cooling of them is necessary. This last point is very important at my furnaces, as I see that at the Roechling furnace about $2,500 is spent for ventilators.

The speaker mentions, further, the previously well-known remedy to better the $\cos \phi$ by the arrangement of an overexcited synchronous motor. It is likely that this in a few cases may be plausible, but it ought to be considered closely in every case

whether this would be practicable and cheaper, and it must be especially noted that the effect of the furnace by this method will not be improved to any extent.

The speaker, further, mentions the low figure for the power, and that this will limit the furnace in question to hot charges only. This is distinctly pointed out in my paper as the case. This figure will, of course, to a great extent be increased when cold stock is to be treated. I have already thought that the figure put down for the power would have to be increased, and I refer in this respect to the table in Fig. 8 (all electrical calculations were made by Messrs. Nissen and von Krogh).

In my furnace no fans for artificial blowing are used, and the whole furnace is tilted independently of the magnet. The $\cos\phi$ is as favorable as can be imagined without complication of auxiliary secondaries. Tenders from works here show that such a 30-ton furnace can be sold (f. o. b. works, and without lining) for about $40,000. On comparison, it can be stated that a Roechling-Rodenhauser furnace for a 5-ton charge will cost about $27,000.

Mr. M. Unger (*Communicated*): While the 30-ton induction furnace proposed in this paper may operate with a power factor of 50 percent, such a low frequency is required that the power installation will be very costly.

Attention is therefore called to the possibility of building induction furnaces of unlimited capacities for operation on commercial frequencies and with high power factor. This object is gained by combining a number of smaller units into one furnace. The advantage of so doing is apparent when it is recalled that the smaller a furnace is, or the smaller the charge in a given furnace is, the higher will be the power factor at which it will operate.

This combination of units may be considered equivalent to the use of a number of electrodes in parallel in each phase, as used in large arc furnaces.

From an electric point of view, it is therefore possible to build induction furnaces of any size to operate with high power factor on ordinary frequencies. The cost and metallurgical requirements will of course be the limiting features.

With reference to the furnace under consideration, I would

suggest that it may be of advantage to increase the number of secondary circuits from 3 to 4 or 6. In so doing, the shape of the crucible will be more complicated, but that may not be so serious. See Fig. 9.

Based upon the estimate given in the paper it will be found that if the 30-ton furnace was operated with a charge of 22½ tons, that is 7½ tons instead of 10 tons per ring, the power factor would increase from 50 percent to 61 percent. Consequently, a 30-ton, 4-ring, 2-phase furnace, as indicated in the attached sketch, would operate with a power factor of 61 percent, the frequency being 8 cycles per second, or if we use 12½ cycles the power factor would be about 44 percent. It will be

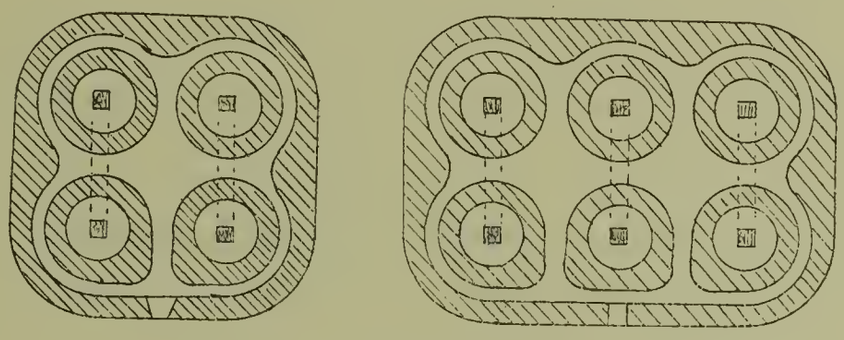


Fig. 9.

noted that the radiating surfaces of the 3-ring and 4-ring furnace would be about equal.

Similar estimates for a 30-ton, 6-ring, 3-phase furnace shows that at 8 cycles it would operate with a power factor of 75 per cent, and at 12½ cycles with a power factor of 59 percent. This is under the assumption that the same diameters and spaces between the primary and secondary rings are maintained as in the proposed 30-ton, 3-ring furnace. In a furnace with suitable proportions, the power factor would perhaps be somewhat lower, but it seems that a 30-ton, 6-ring, 3-phase furnace, operated at 15 cycles, may have a power factor of 50 percent. The cost of such a furnace will no doubt be somewhat higher than of the proposed one, but the saving in the power equipment will be very appreciable.

Mr. Carl Hering (*Communicated*): To take a single step from a 5-ton to a 30-ton furnace (the original announcement of the title of the paper said it was to be one of 50 tons) is not

only a large one, as the author states, but in my opinion, also a bold one, and the propriety of such a large step may be questioned.

Mr. Hiorth's calculations bring out the fact that the power factor diminishes as the size increases, and to counteract this in part, the frequency must be diminished; he has reduced the latter to 8, which is extremely low, and in spite of this his estimated power factor is only 50 percent. This shows that as far as these factors are concerned, the principle of the induction furnace is less well adapted to large than to small sizes.

His chief basic figure rests entirely on the assumption that under normal conditions the heat loss, that is, the heat flow from the molten metal to the outside, depends only on the outside surfaces of the crucible, and is approximately a constant per square meter of such surface, which constant is assumed to be the same in the large as in the small furnace. From his own figures I find this constant to be 3.3 kw. per square meter, or 0.30 per square foot, while just enough current is passed to keep the temperature constant, and while no refining or melting is taking place. This therefore represents the continuous loss through the crucible walls and the top, and constitutes the entire heat energy developed in the metal.

It would seem to be reasonable to expect a better thermal insulation per square foot, on the sides of the troughs for the large than for the small furnace; but on the other hand, at the top and bottom surfaces, it would probably be worse for the large one than for the small one, on account of the very much larger masses of copper there (13.5 tons as compared with 1.75) which seems to be necessitated partly by the poorer power factor.

It seems to me to be more rational to apply this reasoning to the outside surfaces of the metal rather than the crucible; in this way a valuable lesson can be learned. Assuming, for want of better data, that the radiation loss from the top surface, per square foot, is the same as the conduction loss per square foot on the sides and bottom, then I find that the mean value of this constant loss is 13.9 amperes per square meter of this metal surface; this equals 1.2 kw. per square foot. From this it follows by the same reasoning that, for the condition of least possible metal surface for a given volume of molten metal, namely if the

hearth were a hemisphere, the heat required to keep the metal at the refining temperature would be only about 6.6 kw. per ton for a 30-ton furnace and 12 for a 5-ton furnace, instead of 12 and 36, as in the furnaces described in the paper.

This is very instructive as it indicates the great reduction in power which could be obtained in such furnaces in which it is possible to make the hearth approach the theoretically best form, instead of being in the form of relatively long and narrow channels with very much greater surfaces, as they are in induction furnaces.

This very great possible heat economy is due to the fact that the heat loss through the walls per square foot, is quite great, hence a reduction of the number of square feet becomes a very important factor in reducing these losses.

I have discussed this more fully and have given further details, deductions and confirmations of these figures in an article in the November issue of *Metallurgical and Chemical Engineering*.

The resistivity of molten steel which he obtained, namely 1.4 ohms per square millimeter and meter length, agrees fairly well with the values I compiled from various sources in these Transactions, Vol. 15, 1909, p. 223, the slightly lower than the mean of some of these, namely 1.7. It is appreciably lower than some crude determinations which I made recently.

DR. G. B. WATERHOUSE: I cannot pretend to discuss this paper, but I wondered if some one, for instance, Mr. FitzGerald, could tell us very briefly what advantage the 30-ton induction furnace offered over such types as the Roechling-Rodenhauser or the Girod furnaces, which are already made up to 30 tons capacity.

MR. F. A. J. FITZGERALD: I really do not know, Mr. President, what advantage it would have. I have done some work on the induction furnace, and I have seen the Roechling-Rodenhauser furnace working, and there is no question in my mind at all but that the Roechling-Rodenhauser is enormously superior to any of the various other induction furnaces.

PRESIDENT WHITNEY: You are not comparing them with the types of arc furnaces?

MR. FITZGERALD: No.

MR. CARL HERING: Carrying out this same idea of calculating

the power from the losses through the walls, I endeavored to figure out roughly from this paper what the minimum possible power would be if a furnace be made so that it had the least possible exposed metal surface. Such a furnace, of course, would be a sphere, but that is impracticable, and the nearest practicable form is a furnace with a charge in the form of a hemisphere. A furnace constructed in that way would give the least consumption possible per ton of iron, to keep the iron at a certain temperature. Unfortunately the manuscript copy of this paper, which I used for this purpose, was not clear. I will now revise the figures and hope to publish them soon. The deductions appear to give some very interesting results.

A paper read by title at the Twentieth General Meeting of the American Electrochemical Society, in Toronto, Canada, September 21, 1911, President W. R. Whitney in the Chair.

ELECTRIC FURNACE TREATMENT OF NICKEL ORE, AND THE DEVELOPMENT OF A COMMERCIAL PROCESS.

By Walter Leonard Morrison.

(A Thesis presented to Case School of Applied Science for the degree of Engineer of Mines.)

PREFACE.

This thesis has to do with certain tests of a commercial nature made at the plant of the Consolidated Nickel Company. The power plant at Webster, N. C., on the property of the Consolidated Nickel Company, will be described in conjunction with the furnace. Data as to cost of operation and power consumption will be included.

Sketches and drawings of new electric furnaces will be described, reasons being given for the proposed changes.

The nickel deposits from which the nickel ore comes are owned by the Consolidated Nickel Company.

I am indebted to that company for the privilege of writing this thesis, and including much valuable data. Most of my information regarding the ore deposits was obtained from reports published by the State of North Carolina. I also obtained many suggestions from the papers of the American Electrochemical Society, in the designing of new furnaces.

INTRODUCTION.

Location of deposits.
Altitude.
Roads.
Geology.
Analysis of Ores.
Tonnage.

Development.
History.
Mining.
History of the methods of treatment of the Ore.
Ferro-Nickel-Silicide and its Properties.
Uses of Ferro-Nickel-Silicide.

The nickel deposits are located about one-half mile (1 km.) from Webster, county seat of Jackson County, North Carolina. It is about three miles from Dillsboro, a station on the Murphy branch of the Southern Railroad, which is 48.9 miles (80 km.) from Asheville, N. C. The property is divided into a northern and southern half by the Tuckaseigee River. The altitude of this property is about 2,000 to 2,300 feet (600 to 700 m.) above sea level. The property can be reached by dirt roads from Dillsboro, which parallel the Tuckaseigee River. The property is such that it can be worked the year around.

The nickel deposits are associated with the Peridotites of the Southeastern Appalachians and run approximately north and south for a distance of over 7,000 feet (2,200 m.) and nearly 1,500 feet (450 m.) wide. The north end of the deposit immediately adjoins to the east the town of Webster. The ore is very closely allied to the Garnierite of New Caledonia. It is a hydrous silicate of magnesium and nickel, but variable in composition as regards the mutual displacement of nickel and magnesium. It is amorphous, filling certain cracks with incrustations of delicate, hemispherical, or stalactitic forms, usually soft and friable, falling to pieces in water, unctuous to the touch, and adhering slightly to the tongue. It varies in color from brown through pale yellowish green, to a deep apple green. The depth of color usually accompanies an increase of the nickel contents. These seams or veinlets are from one inch to eighteen inches (2.5 to 45 cm.) in thickness, and are very numerous and traverse the rock in a manner similar to the veins of the human body.

The analysis of ores from the same cross cut vary greatly in chemical composition and color. The elements which appear to vary most are iron, magnesia and alumina. The reddish brown variety of ore carries the greatest amount of iron and alumina, while the green ores carry the greatest amount of magnesia.

Analysis of hard green ore:

Nickel Oxide	1.90 percent
Silica	34.00 "
Magnesia	48.00 "
Alumina	3.40 "
Iron Oxide	1.00 "
Chromic Oxide	1.70 "
Water (of crystallization)	10.00 "

Analysis of the soft green variety (Genthite):

Silica	42.00 percent
Nickel Oxide	8.10 "
Magnesia	36.20 "
Water (of crystallization)	10.80 "
Alumina	1.20 "
Ferric Oxide	1.00 "
Chromic Oxide	2.10 "

Analysis of Genthite from Webster (Geol. Survey of Canada):

	No. 1	No. 2
Silica	49.89	55.38
Nickel Oxide	16.60	17.84
Magnesia	22.35	15.62
Water (of crystallization)	12.36	10.77
Alumina	0.00	0.00
Iron Oxide	0.06	0.56
Cobalt Oxide	0.00	0.00

The quantity of ore available running higher than 10 percent nickel oxide is indeed very small.

The tonnage of the ore (averaging 1.75 percent nickel is so enormous that it is almost impossible to give a close approximate of the total quantity. The depth of the deposit has been investigated by diamond drilling, and has been proven to a depth of 165 feet (50 m.). Although as shown some of the ore contains from 16 to 17 percent of nickel oxide, no very large amount of similarly rich material could be secured in mining. Most of the so-called rich seams are occupied by greenish nickel hydrosilicate which will assay 5 to 7 percent nickel oxide. Much of this secondary vein matter is a low grade nickel ore, and will assay 1.50 to 3 percent nickel oxide. This ore also carries a small amount of cobalt oxide. These nickel deposits are found in intimate association with a mass of peridotites, made up princi-

pally of two varieties, which are doubtless differentiates of the same magma, viz.: Dunite, made up entirely of olivine and chromite; Websterite, composed essentially of bright green diopside and pale green bronzite. The development work undertaken consists of a series of parallel trenches or ditches, to a depth in some cases of 30 feet (9 m.). These trenches are designed to give an idea of the relative abundance and disposition of the veins of silicate material. Shafts have been sunk in some cases to depth of 90 feet (28 m.).

This deposit has been known since the latter part of the 60's, and from time to time efforts have been made to develop the property and bring it to a working basis, but being low grade ore it has been impossible to work this property for two reasons: first, because the former owners did not have money enough to develop a low-grade propositon of this magnitude, and second, that until recently there was no process that could treat commercially the ore without hand picking. Different processes have been tried from time to time but without any promising results, for working the property as a whole. If, however, the ore was mined and the high-grade seams separated, there is no question but that the property could have been worked before, as enough 3 and 4 percent ore could have been mined and shipped to those plants which are using the New Caledonia ore. This ore as well as the New Caledonia ore is free from sulphur and copper, and this makes it a very desirable one for treatment in conjunction with arsenic ores, and ores high in sulphur. To expel from the latter the sulphur and arsenic, they must be roasted in heaps or furnaces, which is expensive and tedious, and in most cases ties up thousands of dollars worth of ore in the roasting heaps for months before the commercial article can be put on the market. To extract the nickel is also a tedious and complicated process, which with the Webster ore can be entirely eliminated.

The nature and extent of the deposit make the mining a very simple matter. No tunnels or ore shafts; on the other hand the ore for the most part can be handled with a steam shovel and can be put into cars at a cost not exceeding 11 cents per ton. Even if mined by hand and with the present price of labor, the cost of mining and putting into cars would not exceed 38 cents per ton.

As stated above, attempts were made to reduce the nickel from

this ore by well-known processes, but difficulties of one sort or another always arose. To smelt this ore like the New Caledonia ore, and convert it into the sulphide, the average ore would have to be hand-picked, and then the difficulty would arise to get suitable sulphur-carrying material to the mines at a price which would permit successful smelting on the ground.

Wet methods were also tried to extract the nickel, which were very promising till tried on a larger scale. While it was possible to extract over 90 percent of the nickel contents by leaching, trouble arose in the filtering due in part to the ore being so high in magnesia and sliming so very badly, and for that reason these methods did not prove sufficiently promising to warrant the installation of a plant.

Since the electric furnace has proven its commercial value, new attempts have been made to extract the nickel from this ore and make a commercial product. As the largest part of the nickel consumed is used in connection with the manufacture of steel, it was decided to make an alloy of nickel which could be successfully used in the manufacture and refining of steel. The results obtained were very satisfactory, and by one step a marketable product is made from the crude ore.

The nickel as reduced in the electric furnace from the ore contains iron, silicon, nickel, magnesium, aluminum and chromium. The alloy is a complex silicide of iron, nickel and the other metals. This silicide is silver white in appearance, showing a fine grain on the fracture. When the nickel content reaches 20 to 25 percent the metal has vertical striations.

Analysis of various samples of the silicide show the contents to be about as follows:

Nickel	10 to 25 percent
Silicon	30 to 20 "
Iron	55 to 50 "
Other Elements	5 "

The silicon, aluminum and magnesium act as scavengers and gas removers. The effect of nickel and chrom in steel is well known. One hundred parts of ore will produce about 20 parts of the silicide.

EXPERIMENTAL INVESTIGATIONS.

I. Reduction of Ore by Reducing Gases at a High Temperature.

II. Reduction of Ore by Semi-fusion.

III. The Wet Processes:

(*a*) *Sulphuric acid.*
(*b*) *Ammonium chloride.*
(*c*) *Sodium chloride.*
(*d*) *Sodium sulphate.*
(*e*) *Chlorine gas.*

IV. The Laboratory Electric Furnace.

(*a*) *Design.*
(*b*) *Operation.*
(*c*) *Capacity.*
(*d*) *Power.*

V. The Electric Furnace Products.

(*a*) *Slag and reactions.*
(*b*) *Metallic products.*
(*c*) *Analysis of slag.*
(*d*) *Analysis of silicide.*
(*e*) *Analysis of ore.*

VI. The Commercial Electric Smelter:

(*a*) *Location.*
(*b*) *General plan.*
(*c*) *Apparatus.*
(*d*) *Design of the furnace.*
(*e*) *Construction of the furnace.*
(*f*) *Operation of the furnace.*
(*g*) *Electrode trouble.*
(*h*) *Dynamo trouble.*
(*i*) *Trouble with ore.*
(*j*) *Mining and tramming.*
(*k*) *Efficiency of the furnace.*
(*l*) *Cost of operation.*
(*m*) *Output of plant.*

VII. CONCLUSIONS.

(*a*) *The shaft furnace.*
(*b*) *The crucible furnace.*
(*c*) *Electric smelter design.*
(*d*) *Estimates on the cost of installation of* 100-*ton electric smelting plant.*
(*e*) *Estimate on the cost of operation of* 100-*ton electric smelting plant.*
(*f*) *General remarks.*

I.

REDUCTION OF THE ORE BY REDUCING GASES AT A HIGH TEMPERATURE.

In our experiments it was thought that perhaps our ore might be reduced directly by hot CO. For the experiment I used a

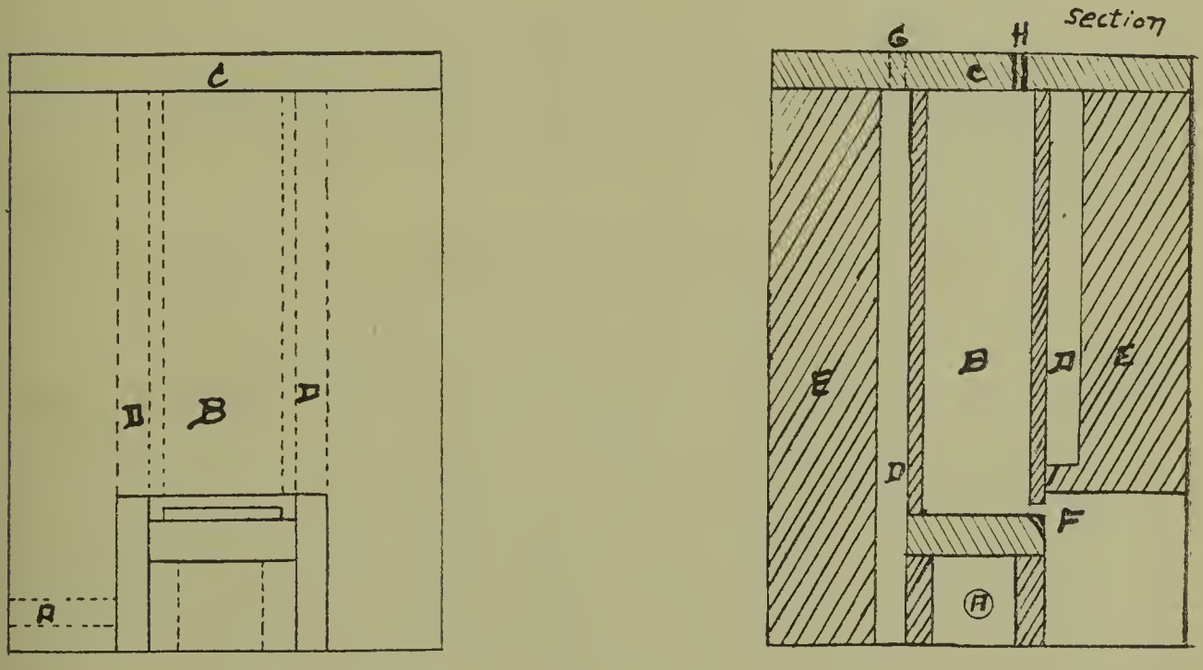


FIG. 1.

furnace as shown in fig. 1. It consists of an upright flue tile eight inches (20 cm.) square, which holds the ore mixture (B). A two-inch (5 cm.) air space (D) was left around the tile for heating. A blast lamp of the Buffalo Dental type was introduced at (A) and furnished the heat. The furnace walls (E) were of fire brick, eight inches (20 cm.) in thickness. (G) is an outlet

for waste gases. (H) is an outlet for the gases from the ore mixture. The ore was introduced at top of tile, then the cover sealed on with cement.

The ore used for these experiments with CO was the green micaceous variety, very soft and very fine, when moist it is quite plastic. Charcoal was placed in the bottom of furnace near the opening (F), then the ore on top mixed with charcoal.

Heat was applied gently for three or four hours till the ore mass reached a fairly uniform temperature of 350° C. (a Hoskins thermal couple and millivoltmeter was used for all temperature determinations above 300°C.). Two graphite crucibles were used as condensers over the opening (H). The crucibles were placed top to top, with a hole in the bottom of each for the admission and outlet of gases. A piece of cheese cloth, wetted, was used between the crucibles to aid in the condensation. The charcoal at (F) was ignited by a Bunsen burner. Considerable moisture was driven from the ore at first. The heat was very slowly raised and a new cheese cloth placed between the crucibles when the ore mass reached 400° C. Then at 450° C. and 500° C. the cloths were changed. The temperature of the ore mass was now raised till it reached 1100° C.; it was held here for about an hour then allowed to cool.

The cheese cloths were each placed in a porcelain crucible dried and treated with concentrated nitric acid to test for nickel to see if any had formed volatile nickel-carbonyl and been volatilized from the ore. Not a trace was found from any of the cloths, or in the graphite crucibles. The ore was then examined, and although the color was changed the ore appeared the same. Samples were run for nickel contents, and allowing for water of crystallization, the analysis tallied very closely with that of the original ore. The ore did not appear to be the least bit lumpy, showing a tendency to fuse. This ore mixture after being removed from the furnace was passed over a Wilfley concentrating table. The concentrates analyzed about the same as the tails, showing no reduction to metallic nickel. This method was then abandoned, for the nickel appears to be held very strongly combined as the double silicate of magnesia and not to be reduced or affected in any way by the hot carbonous oxide gas.

II.

REDUCTION OF THE ORE BY SEMI-FUSION.

About 60 to 75 percent of the nickel ore runs very high in magnesia (25 to 38 percent) but low in iron. The fact that the ore is so high in magnesia makes it a difficult proposition to form a fluid slag, even in the electric furnace, which produces the highest temperature of any present commercial furnace. The idea naturally suggested itself, that the ore could be subjected to a high temperature, in a reducing atmosphere, and intimately mixed with charcoal, and then removed as soon as reduction fusion had taken place. The semi-fused mass could then be crushed and the nickel separated from the slag by a magnetic separator, or on the Wilfley concentrator.

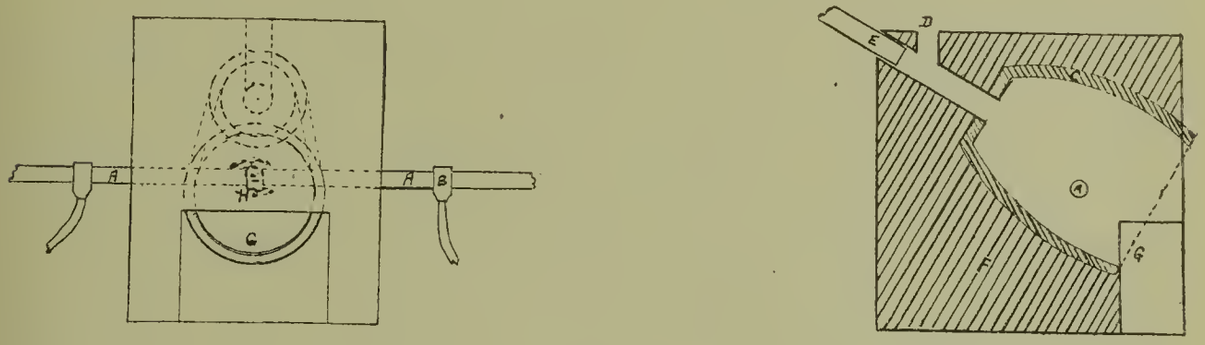


FIG. 2.

For this experiment the furnace shown in Fig. 2 was used. The two electrodes (A) are of 1½-inch (4 cm.) carbon (National Carbon make), and enter the furnace from opposite sides. The furnace proper, or heating chamber, consists of a No. 50 graphite crucible, placed as shown in Fig. 2. The idea was to give the heating chamber floor such a pitch as to enable the slag to flow out at (G). The furnace was set on an iron plate so as to enable me to change the slope of the furnace floor. The arc was situated about 1.5 inches (4 cm.) below the center line of the furnace. The openings in the side of the crucible for the electrodes was sufficiently large to eliminate any possibility of a short circuit with the crucible under ordinary conditions. Immediately surrounding the graphite crucible was a magnesite lining, this was followed by fire brick on the exterior.

The crucible had 1.5 inch (4 cm.) opening in the bottom for

the introduction of ore and fluxes (D). The ram (E) is used in pushing the ore forward, also used to close the opening, stopping the ore flow into the furnace, and escape of the gases upward. The electrodes are secured in a holder which is given a longitudinal motion by means of screw and gears. For these experiments a reddish brown variety of nickel ore was used, which is easily fluxed with lime.

The charge used was as follows:

Ore	100 Parts
Lime	20 "
Coke	15 "

The arc was started in the furnace using direct current, 300 amperes at 50 volts. After the furnace attained a good white-heat, ore was slowly fed in, at first the ore flowed through without fusion, but as soon as fusion took place the rush of ore ceased. The slag flowed from the furnace in a continual stream, and things were going nicely for a few hours; then trouble began. The ore being very fine, dusted considerably, and the dust in a semi-fused state hung to the sides of fusion chamber; finally this semi-fused mass caused a short circuit of the current between the electrodes and the crucible. The current had to be shut off and the furnace allowed to cool. The semi-fused slag was crushed and concentrated on the Wilfley table. The amount of ore treated per kw.-day was very low (14 pounds) compared to the results of a fusion or slag-making electric furnace, which will smelt 23 to 27 pounds of ore per kw.-day. The concentrates from the Wilfley table were high in nickel, but the total extraction of nickel from the ore was about 60 percent, which is very low.

In order to eliminate the danger of the short circuit, a magnesite lining was used, in a furnace as shown like the Moissan enclosed-arc furnace.[1] During the run this furnace became overheated, and even the magnesite on the sides and top fused. The slag came away very easily, but the amount of ore treated per kw.-day was even smaller than in the other case, because of the amount of heat that must necessarily be wasted by radiation on account of the arc being outside of the ore.

[1] This furnace is illustrated and described on page 141 of Eimer and Amend's general catalog.

In the case of other arc furnaces, the ore absorbs heat all around the arc. A furnace of the type just described would, after being run for a few hours become so strongly heated that the lining would be in serious danger of falling in, from fusion. The experiments along this line were discontinued, for they did not appear very promising.

III.

THE WET PROCESSES.

For these tests, soft green ore was used carrying 3.36 percent nickel.

(*a*) *Sulphuric acid.*—Using a 10 percent solution of sulphuric acid on the raw, dried or roasted ore, hot or cold, the maximum extraction of the nickel obtained was 29 percent—an unsatisfactory result. The same ore was next heated in a crucible with charcoal to 900° C. for two hours, and then leached one to five hours with five times its weight of 5 to 30 percent H_2SO_4. The extractions varied from 20 to 70 percent of the nickel present, according to the strength of acid and time of treatment. Seventy-five percent seems to be the maximum extraction to be expected by sulphuric acid treatment and to get this would require at least one ton of acid per ton of ore; not over 25 percent of this acid could be recovered. An estimate of cost of treatment is $15.05 per ton, with $11.25 as the value of the nickel recovered.

(*b*) *Ammonium chloride.*—Several tests were made heating the ore with this salt, to break up the silicates, and then treating with acid. There appeared to be no resultant advantage.

(*c*) *Sodium chloride.*—The ore was heated with this salt and charcoal, to decompose the silicates, and then leached with acid. The extraction by acid was not increased.

(*d*) *Sodium sulphate.*—This salt and charcoal were used similarly to the above, but with no influence on the extraction of the nickel.

(*e*) *Chlorine gas.*—This gas was passed through dry and through moist ore, but did not decompose the silicates.

IV.

THE LABORATORY ELECTRIC FURNACE.

(*a*) *Design.*—The first furnace used in connection with this work of smelting refractory ores was a Borchers furnace obtained from Eimer and Amend. Capacity 100 amperes at 50 volts. The lower electrode is insulated from the furnace shell with mica and asbestos, and extends thru the bottom of the furnace; this electrode is fixed. The upper electrode, a 1,25-inch (3 cm.) carbon is movable, and is controlled by a screw thru the electrode holder. The upper electrode is insulated from the furnace proper by asbestos at the two supports of the electrode control.

The furnace shell is made of sheet iron and angle iron. It is lined with first a layer of asbestos then a layer of burned lime. The interior is a solid mass of magnesite from which the crucible has been cut. There is a tap hole for removing the molten mass. This furnace was not a satisfactory one for semi-commercial demonstrations. The crucible was very small to begin with, and would hold very little molten material. Then too it would freeze up very readily, and would have to be taken apart. For fusions not requiring any great length of time the furnace did very well, but after operating an hour or two the trouble would begin.

A larger furnace was then built using a larger electrode, thus allowing more current to flow, consequently more heat was generated. It is made of ⅛-inch (0.3 cm.) iron plate and angle irons. It is box shape, 18-in. square by 18-in. (45 x 45 cm.) high. The top electrode is controlled about the same as in the Borchers furnace, except that it has a bevelled gear on top of the screw engaging a gear to which is fastened a shaft and small hand wheel. This allows the operator to adjust the electrode without being over the furnace. The bottom electrode is a 6-in. (15cm.) Acheson graphite electrode, which is secured and insulated same as that in the Borchers furnace, it comes within 3 in. (7.5 cm.) of bottom of the crucible. On top of the electrode and part way up the sides of the furnace is a coke lining, using pitch as a binder. The coke is placed in the bottom of the furnace to retard conduction of heat thru the graphite, which is an excellent conductor of heat as well as of electricity.

The reason for using this graphite electrode is that it was the only thing available, it having been purchased for another use. The inside of the furnace shell was lined with ¼-in. (0.6 cm.) asbestos, then built up with fire brick, leaving a crucible in the center of the furnace, 13 in. (33 cm.) deep by 8 in. (20 cm.) diameter. A cover for the furnace was made by clamping several fire bricks together, leaving an opening for the electrode and a vent for the escaping gases. The movable electrode was of carbon 2¼ in. (5.5 cm.) diameter by 48 in. (120 cm.) long, made by the National Carbon Company.

(*b*) *Operation.*—When starting the furnace, a little powdered coke was usually placed in the bottom of the furnace to avoid a short circuit on the dynamo. As soon as the arc was fairly continuous the ore was fed in around it. Soon a slag would be formed; then the furnace would draw a steady load with very slight fluctuations of current. We averaged a tapping of the furnace about every 15 to 20 minutes, and would get about 10 lb. (4.5 kg.) of slag and metal, about a quart (litre) in all. The question naturally asked at this point would be, is it necessary to tap so often? My experience has been that it is; and for two reasons, first, that the efficiency of your furnace is increased if you remove the slag as fast as it has become inactive in the reactions within the furnace; second, if the "source of heat" which is near the dividing line of the slag and ore be raised too much above the bottom of the furnace, the metal on the bottom would be cooled to its solidifying point, and the furnace will start to freeze; after working a few hours in this way the slag begins to freeze around the tap hole, then trouble with the tapping begins which usually ends in a premature shut-down.

I have also noticed that if the furnace is let run too long between tappings, carbides are formed in the slag, which renders it very infusible and sticky, so that it will not flow. The formation of carbides and the part they play in the furnace reactions, I will treat of later. When tapping the small furnace, the current would usually drop to zero, and the electrode would have to be lowered, but there was always enough semi-molten material in the bottom of the furnace to make an electric circuit without causing any trouble. The furnace was kept full of ore, the gases escaping very freely. The ore being very fine, there was con-

siderable dusting; a 5-in. (12.5 cm.) flue was placed over the vent for escaping gases, which carried the fine dust and fumes away.

(*c*) *Capacity.*—The capacity of the furnace was limited to the capacity of the gas engine, which gave a maximum of 15 kw. at the switchboard. The furnace would treat about 100 lb. (45 kg.) of ore in 6 hours. The runs with this furnace were comparatively short, the longest one being ten hours.

(*d*) *Power.*—The electric power was derived from a 60 kw.-D. C. generator, compound wound, 125 volts, 480 amperes. The generator was belt-connected to a 40 h. p. gas engine. The gas engine was a one cylinder, four cycle, 24 in. (60 cm.) stroke; made by the Springfield Gas Engine Company. A high resistance was placed in the shunt field of the dynamo in order to lower the voltage to 50 or 60 volts, which is as high as a furnace of this type ought to run on. It is rather poor economy to run a dynamo of this kind at such a low voltage, on account of the low efficiency. I do not think the efficiency was over 60 percent. We utilized such apparatus as we had for this work, and since it was for a short time only, a low efficiency did not matter. The current used by the furnace varied from 350 to 450 amperes. The conductor leads between the switch-board and the furnace consisted of two 500,000 circular mils (250 sq. mm.) cables, one attached to each electrode. The dynamo was protected from short circuits by fuses and a circuit-breaker. The readings of the voltmeter were taken from the bus bars at the switch board. The ammeter was of the shunt type.

Several charges of the green and brown ores were heated with charcoal in graphite crucibles in a gas blast furnace, in which a temperature of 1550° was obtained.

No.	Wt. of Brown Ore	Wt. of Green Ore	Wt. of Charcoal	Condition of Slag
1	90	10	5	Slag very fluid
2	80	20	5	Slag very fluid
3	70	30	5	Fusion incomplete
4	60	40	5	Fusion incomplete
5	50	50	5	Fusion incomplete
6	40	60	5	Slag glassy
7	30	70	5	Fusion incomplete
8	20	80	5	Stony slag
9	10	90	5	Stony slag

These various quantities of ores were run so as to give some idea as to their self-fluxing qualities. No. 6 appeared to give the best slag. Since the greater part of the ore is high in magnesia (the green ore) it is an advantage to be able to use the brown ore as flux instead of barren matter.

Various runs made in the laboratory with the laboratory electric furnace, checked very closely with those made in the commercial electric furnace, *i. e.*, as regards the amount of ore smelted and metal produced, per kw.-day.

V.

THE ELECTRIC FURNACE PRODUCTS.

(*a*) *Slag and reactions.*—In the treatment of these ores, the slag question was at first very difficult to handle owing to the extremely high percent of magnesia, and low iron, calcium, and alumina content. The alkalies in the ore are quite high, on account of the large quantity of mica. Several attempts were made to treat the hard green ore alone with various fluxes in the electric furnace. Fusion took place, but the slag was quite viscid and would solidify almost as soon as it met the air, which naturally caused considerable trouble around the tap hole. Then too, the metal would not separate and collect at the bottom of the furnace unless the slag was quite fluid. The brown ore which is quite high in iron and alumina but low in magnesia is an excellent flux for the high magnesia ores. Our slags under good working conditions varied about as follows:

Silica	40.00	to 45.00	percent
Alumina	30.00	to 20.00	"
Ferric oxide	2.00	to 0.50	"
Calcium oxide	3.00	to 10.00	"
Magnesia	20.00	to 15.00	"
Nickel oxide	0.40	to 0.80	"
Alkalies	4.60	to 8.70	"

The following are percentage analyses of slag:

SiO_2	Fe_2O_3	Al_2O_3	CaO	MgO	Ni
43.20	...	24.60	16.80	12.20	0.40
44.00	2.00	25.40	14.00	12.60	1.12
42.00	0.85	21.80	4.20	18.00	0.43
44.00	2.00	29.20	14.00	16.00	0.40
42.40	0.85	25.40	8.40	12.60	0.63
41.60	1.00	29.60	8.60	16.10	0.53

All the above slags gave little or no trouble in the furnace,

flowed freely and allowed the metal to separate and collect in the bottom of the slag pot.

In the smelting of this ore in an open furnace, there was considerable loss of nickel by volatilization. The alkalies and magnesia also volatilized to a considerable extent. The loss by volatilization is from 15 to 20 percent of the weight of ore.

I have noticed in connection with the operation of the furnace upon these ores, that lime in conjunction with carbon up to certain limits plays a very important part in the amount of silicon reduced. I think that the formation of calcium carbide acts as a very powerful reducing agent and breaks up the silicates, liberating silicon and forming more complex silicates.

The iron aids in the collecting of the metallic nickel, very little iron goes to the slag. At first considerable trouble was experienced with the coke, it would accumulate in the furnace and finally cause it to freeze up. This trouble was practically eliminated by using finer coke, instead of ½-in. (1.2 cm.) coke we used 10-mesh (0.25cm.) coke, which gave better results. In order to get complete reduction and a good extraction it is necessary to add a little excess of coke, and being in the fine condition it is easily carried from the furnace with the slag.

The formation of carbides in the slag is a signal that the furnace will soon give trouble, unless the charge is changed or the carbon lessened. The carbides are easily detected by the odor of acetylene about the cool slag. A slag which decomposes as it cools is another indication that something is wrong with the fluxing. The furnace must be tapped at comparatively short intervals of not more than 30 to 40 minutes.

Granulation of the slag was tried and was quite successful; the slag is then easily removed from the metal by simply washing. The metal remains in fairly large pieces, and in the form of shot. On a large scale, granulation would be a very cheap method of separating the metal, and the slag could be easily flushed to the dump.

We tried crushing the slag and concentrating with a Dings Magnetic Separator, but this was not very satisfactory, as very fine particles were so weakly magnetic that they went with the tailings. The Wilfley Concentrator with material thru 30-mesh (0.8 cm.) gave excellent results.

(*b*) *Metallic products.*—The metal reduced from the ore and separated from the slag is in all cases an alloy of nickel, iron and silicon. In no run were we able to obtain a low-silicon alloy, and keep our extraction about 80 percent. This alloy must from necessity carry considerable iron. The amount of metal separated from a ton of ore depends upon the ore treated. The brown ore will give about 440 lb. (200 kg.) of metal to the ton, while the green ore will not give more than 200 lb. (91 kg.) to the ton. Using a mixture of the ores the amount of metal derived, varies from 300 to 350 lb. (136 to 159 kg.) to the ton.

The following are analyses of metal obtained from the brown ores:

Nickel	10.90	11.00	11.50	10.20
Iron	58.30	56.80	57.40	56.90
Silicon	28.20	30.10	26.30	27.40

The following are analyses obtained from green ore:

Nickel	23.95	21.80	22.60	19.30	18.96
Iron	50.70	51.20	48.90	49.10	54.00
Silicon	21.20	18.80	23.24	26.40	22.80

The following are analyses of metal obtained from the mixtures of the ores:

Nickel	15.04	13.70	13.10	13.30	14.80
Iron	54.80	53.90	56.00	57.80	54.70
Silicon	26.60	27.40	25.70	24.00	26.20

In looking over the above analyses, it will be noted that the green ore gives a metal much higher in nickel, all the runs from which this metal was derived were made on picked ore averaging about 6 percent nickel. It is not advisable to pick ore too much, as it will leave a worthless ore which it would not pay to treat; then too the amount of rich ore to the lean ore is about 1 to 10 or 20 and means a large expense to sort out.

(*c*) *Analysis of slag.*—The analysis of the slag was carried out in the ordinary way for silica, iron and alumina, calcium and magnesia. The nickel was determined volumetrically by a modification of the method given in Blair's Analytical Chemistry.

(*d*) *Analysis of silicide.*—This silicide of nickel and iron is insoluble in hydrochloric acid, nitric, sulphuric, or nitro-sulphuric, or aqua regia. It is decomposed by a solution of nitric and a few drops of hydrofluoric acids, when placed on the hot plate. The action of the acids on the finely pulverized metal is very

intense. The quantity and strength of the acids must be just about right to obtain complete solution of the metal in one treatment. About 15 c. c. nitric acid (1-1) and 1 c. c. hydrofluoric is sufficient for 0.5 gram of metal. The complete solution should take place in about two minutes. After solution has taken place, 15 c. c. sulphuric acid is added and the whole is taken down to first white fumes, cooled, and taken up in water. Ammonia is added just short of precipitation, cooled, an excess of standard cyanide solution added, then a slight excess of ammonia (1. c. c.), dilute to 500 c. c., allow to settle and take an aliquot part of clear solution for titration with standard silver nitrate solution. Calculation for nickel, same as in slag.

For the iron determination the metal is taken into solution as in the above cases, taken down to dryness with hydrochloric acid in excess, taken up in hydrochloric acid, diluted with water, the iron reduced with stannous chloride solution, and iron titrated with standard permanganate solution.

For the silicon determination the metal was fused with sodium peroxide in an iron crucible, taken up in water, treated with hydrochloric acid, run down to dryness in an evaporating dish, baked, taken up in hydrochloric acid and water, filtered, and the silicon estimated from the silica in the ordinary way, after treating with hydrofluoric acid.

This ferro-nickel-silicide also contains aluminum, calcium, magnesium in traces, besides 1.5 to 2.5 percent chromium.

(*e*) *Analysis of ore.*—The analysis of the ore was carried out by ordinary laboratory methods. I will enclose several analyses which I made:

Sample No.	Nickel	Iron Oxide	Alumina	Magnesia	Silica	Chromium Oxide
29	0.97	29.00	9.70	1.09	38.50	...
30	1.30	20.00	...	18.60	47.00	1.00
31	1.48	10.00	...	16.30	37.20	2.20
32	1.05	6.80	1.30	22.70	31.40	0.50
33	1.85	7.00	1.30	11.90	42.40	0.80
47	3.35	4.80	7.20	20.70	43.20	...
48	6.00	3.60	6.80	24.50	42.40	...

All the above samples, with the exception of Nos. 47 and 48 were picked at random from different parts of the deposit. Nos. 47 and 48 are some of the picked samples. The highest percent of metallic metal I found in any of the hard green rock was

2 percent, while the highest in the soft green ore was 9 percent nickel. The ore will average 1.75 percent nickel oxide.

VI.

THE COMMERCIAL ELECTRIC SMELTER.

(*a*) *Location.*—The electric smelter and equipment is located on the nickel deposit at its middle point. It is about 400 feet from the river and on an elevation 40 feet above the river at low water mark. The buildings are located on a gently sloping hill which can be seen in the accompanying photograph Fig. 3.

FIG. 3.

(*e*) *General plan.*—The general plan of the buildings is shown in Fig. 4, consisting of:

Boiler room.
Engine and dynamo room.
Furnace room.
Concentrating room.
Storage bins for ore, coke and limestone.

The ore-bins were only put in temporarily, as the plant was started before it was completed. It was originally intended to have a dryer for the ore, so as to utilize the heat of the escaping gases, but it was not installed for these first tests.

(*c*) *Apparatus.*—In the boiler room there are four horizontal-

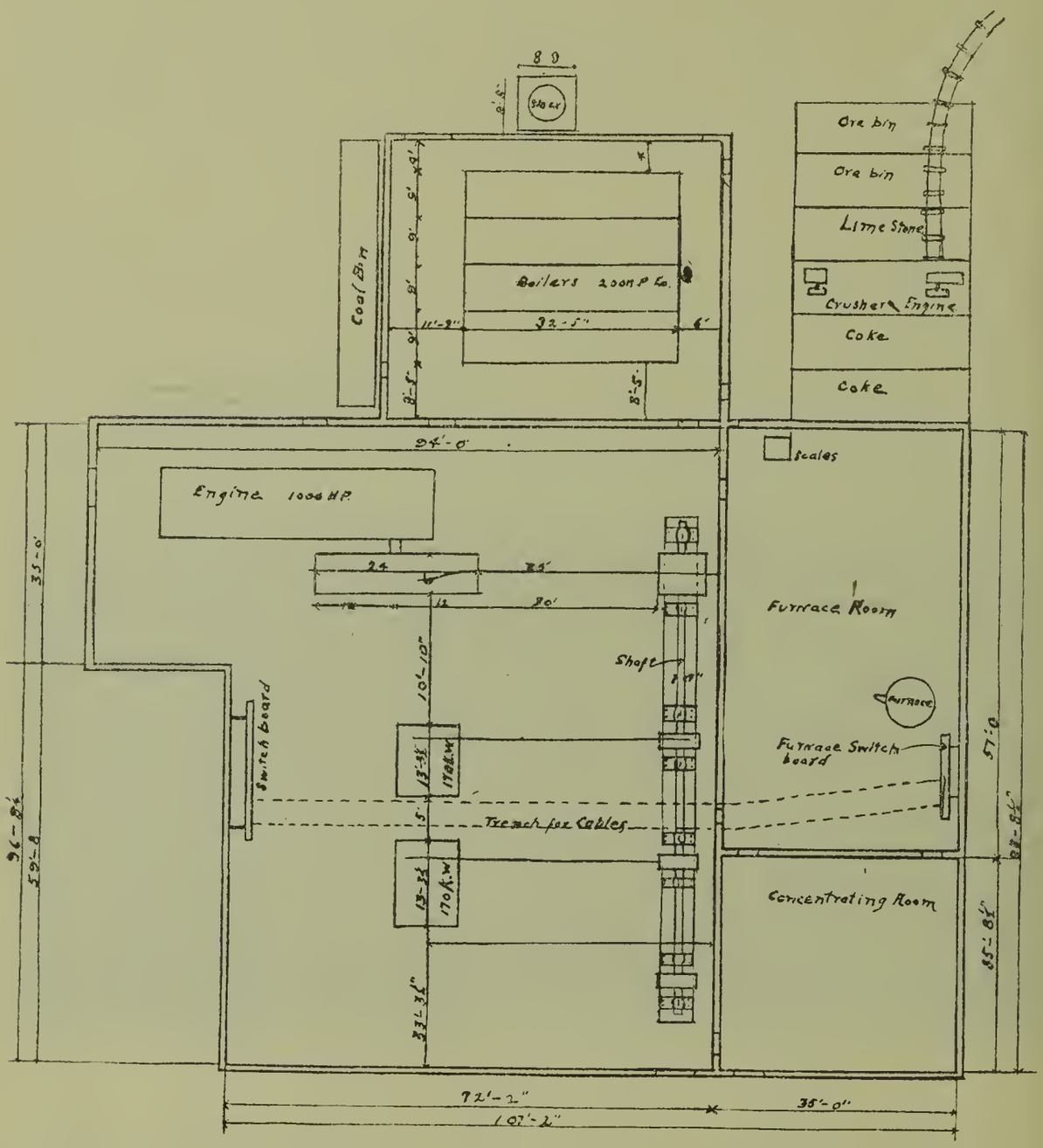


FIG. 4.

fired tube boilers of 200 boiler h. p. each. The fire boxes are of the dutch-oven type, designed to burn wood. However, wood was not easily obtained in the vicinity, so coal had to be burned. Draft was furnished by a 90 foot (28 m.) stack, 6 feet (1.8 m.) in diameter. Feed water was introduced into the boilers by

means of injectors, a reciprocating steam pump was at hand in case of emergencies. Bituminous coal was fired in the boilers; this was obtained from Virginia and Tennessee. A 10-in. (25 cm.) steam main delivered the steam to the engine.

The steam-engine is of the Hamilton Corliss type, single acting, 24-in. (60 cm.) cylinder, and 6-foot (1.8 m.) stroke. Working steam pressure 115 lb. (7.7 atmospheres). Maximum rev. p. m. 75, operating rev. p. m. 70. The fly-wheel is 24 feet (7.4 m.) in diameter, with a 6-foot (1.8 m.) face. The line shaft was driven from the fly-wheel by a 6-foot (1.8 m.) three-ply leather belt. The line shaft drove two D. C. generators which furnished the electric power. Provision was also made for the installation of a third generator.

The two generators were each driven from the line shaft by a 36-in. (90 cm.) 3-ply leather belt. Each generator was of the duplex compound wound type, capacity 170 kw., voltage 50, amperes 3,500; rev. p. m. 375. The two machines were connected in parallel, with an equalizer between. Each machine had eight poles and 112 brushes (area of each brush 3 sq. in. (18 sq. cm.)).

At the switchboard, circuit breakers were installed to protect the machines against an overload. When starting the dynamos a bank resistance was used on the line, so as to get the machines working with constant voltage; after each machine was working with approximately the same voltage, they were thrown in parallel, and the line resistance cut off as soon as the load from the furnace came on.

There are eight 1,000,000 cir. mil. (500 sq. mm.) copper cables on each leg carrying the current between the generator switchboard and the furnace-room-switch-board. These cables are carried thru a concrete conduit below the floor level as shown in Fig. 4 by dotted lines. Each switchboard was equipped with circuit breakers, ammeter and voltmeter. The furnace was connected to the switchboard by eight 500,000 cir. mil. (250 sq. mm.) copper leads to each leg.

In the concentrating room we had two full-size tables set, but they were not used, as the metal separated from the slag very nicely.

The furnace room equipment consists of the furnace and

electrode control, switchboard, 4 slag pots (500 lb. (225 kg.) capacity), and weighing scales for ore and flux. Under the sheds near the ore bins there was a 12 h. p. steam engine, belt connected to a Sturtevant crusher, for crushing coke, limestone and ore.

(*d*) *Design of furnace.*—The furnace used in Webster is

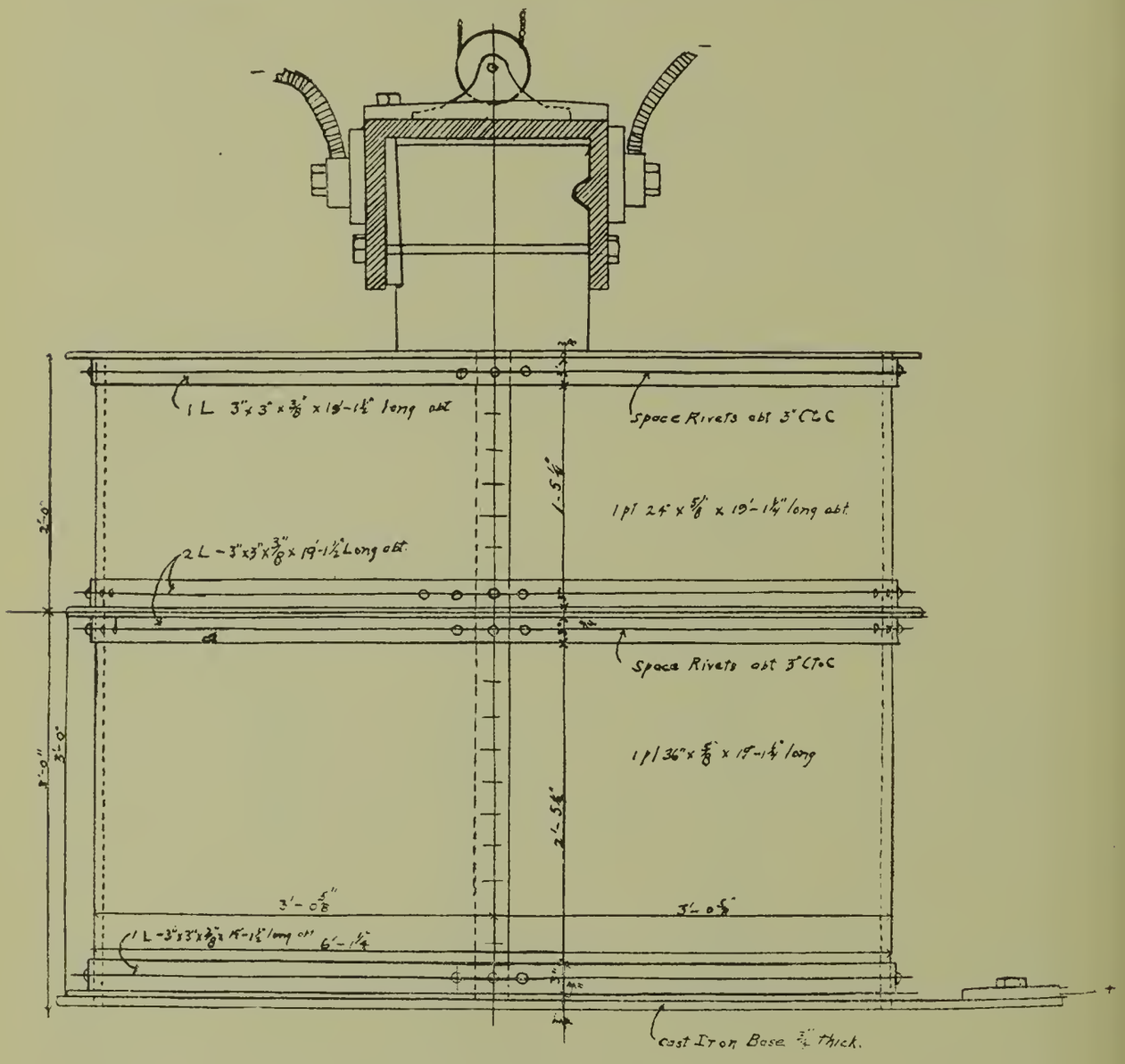

1 L 3" x 3" x 3/8" x 19'-1 1/4" long obt
Space Rivets obt 3" C to C
1 pl 24" x 5/8" x 19'-1 1/4" long obt
2 L - 3" x 3" x 3/8" x 19'-1 1/2" Long obt
Space Rivets obt 3" C to C
1 pl 36" x 5/8" x 19'-1 1/4" long
3'-0"
3'-0"
1 L - 3" x 3" x 3/8" x 19'-1 1/2" long obt
6'-1 1/4"
Cast Iron Base 3/4" Thick.

FIG. 5.

similar to the one which was used at Sault Ste. Marie, by Dr. P. L. T. Heroult, with a few modifications. The furnace was originally designed in two sections, it being intended that the top section could be removed in order to patch up the crucible, but later this was found to be unnecessary.

The bottom plate of the furnace is of cast iron 1.5 in. (3.8 cm.) in thickness, set on porcelain blocks for insulation. Tapered

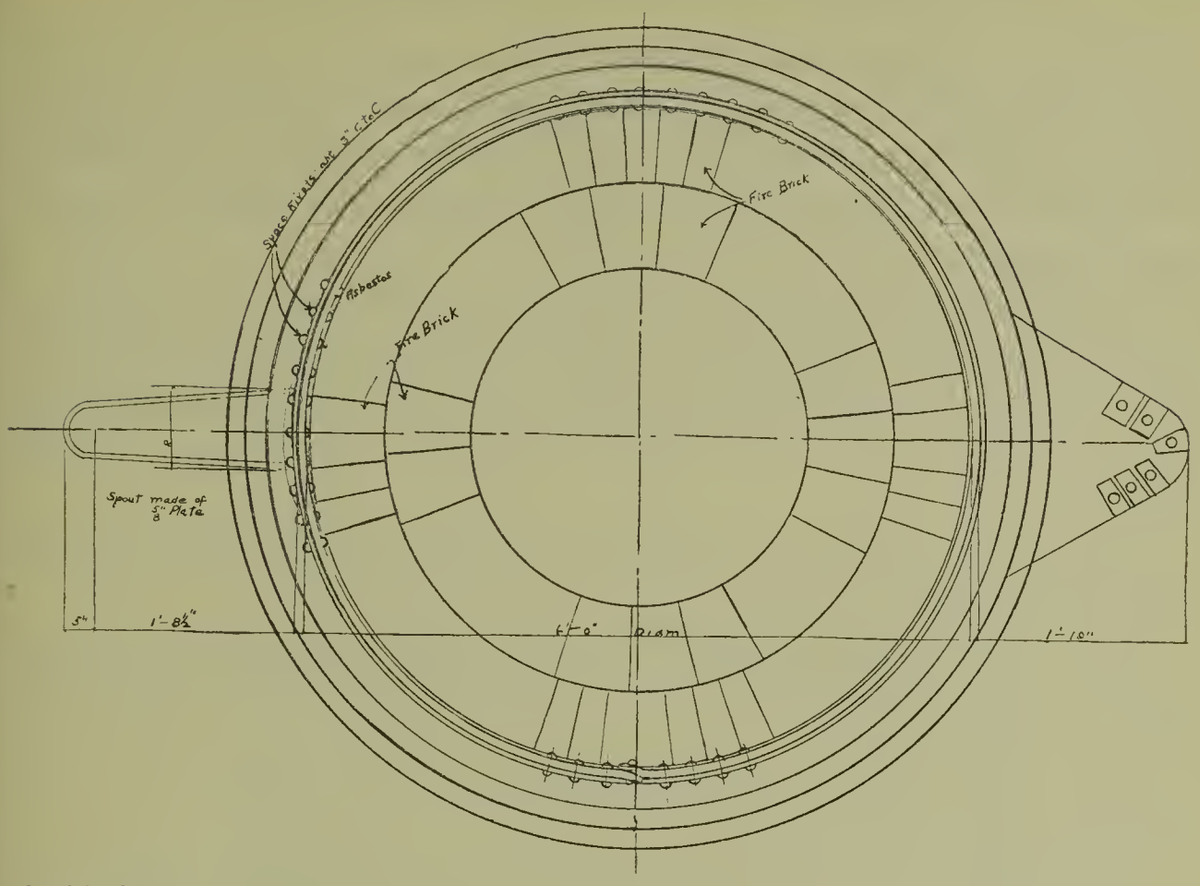

Space Rivets obt 3" C to C
Asbestos
Fire Brick
Fire Brick
Spout made of 5" Plate
5"
1'-8½"
4'-0" Diam
1'-10"

FIG. 6.

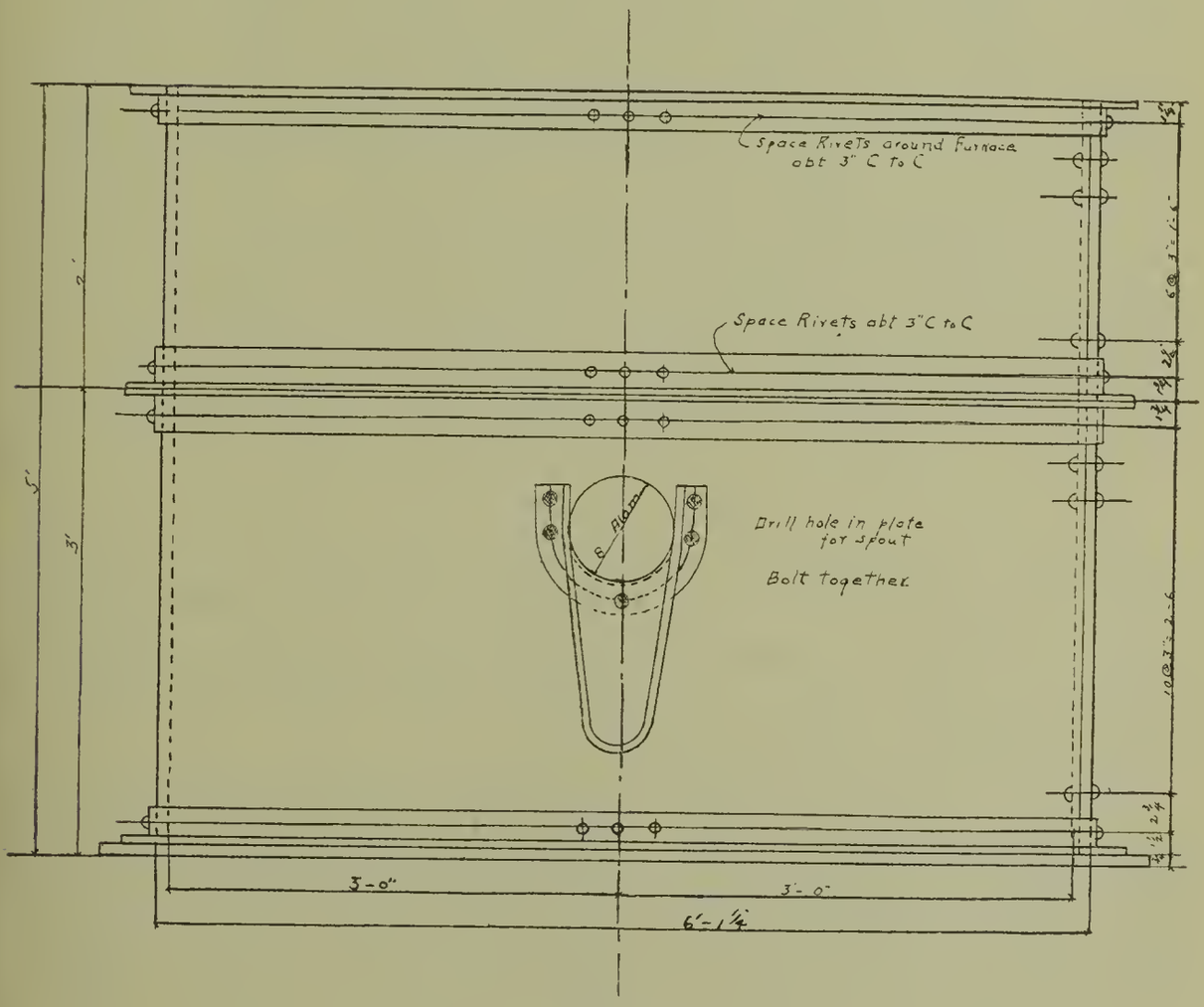

Space Rivets around Furnace obt 3" C to C
Space Rivets obt 3" C to C
Drill hole in plate for spout
Bolt together
8" Diam
3'-0"
3'-0"
6'-1¼"

FIG. 7.

holes were bored in the cast-iron plate, and iron rods 10 in. (25 cm.) long by 0.5 in. (13 mm.) diameter were inserted, in order to give a large area of contact. A large copper plate was fastened to the cast iron at the rear, to which the lugs carrying the cables were secured.

Fig. 5 gives a side view of the furnace, the holder is shown, and method of holding the carbon electrode, also position of cables. The furnace shell is made up of 5/8-in. (16 mm.) iron plate, and 3 in. by 3 in. by 3/8 in. (7.5 x 7.5 x 0.95 cm.) angle irons.

Fig. 6 gives a plan of the furnace, showing the thickness of the fire-brick lining.

Fig. 7 is the front elevation of the furnace, showing slope of hearth and position of spout, one spout being used for both the slag and the metal.

Fig. 8 is a section of the furnace after the lining is in; also showing the covering used.

The shell of the furnace was insulated from the cast iron bottom with heavy asbestos and fibre bushings around the bolts. The interior of the shell was first lined with 1/4-in. (0.6 cm.) asbestos. A mixture of hot graphite and glucose was tamped into bottom of furnace around the iron pegs. Above the pegs the amount of graphite was lessened, and coke dust substituted, and near the top of the bed coke dust with a binder was used. The coke lining was carried up the side for a distance of ten inches; this coke lining appears to resist the corrosive action of the slags better than any other material we used. The cover of the furnace consisted of fire brick clamped together by tie rods.

The lining of the furnace required about four days, one man and helper. The graphite and glucose was heated in an iron soap kettle. Materials used in the lining and cover of the furnace were as follows: One barrel glucose, 3/4 ton coke, 2,000 fire bricks, 1 barrel sand, 1 ton graphite, 1,000 circular fire bricks, 2 barrels fire clay, 20 sheets asbestos.

After the lining was all in, a wood fire was built in the furnace and kept burning three days. This dried the furnace very well. After this the electric current was run in the furnace for several hours, which finished the drying out of the bottom of the furnace.

(*f*) *Operation of furnace.*—The furnace was started with a

little coke in the bottom, but after a few trials this was found to be an unnecessary precaution. The electrode was controlled by a hand-wheel located on the floor, connected to a worm and gear on a carriage, by a chain. To the gear on the carriage was secured a notched wheel which carried a chain connected to the electrode holder. The holder is very heavy being made of cast iron, and weighing about 1,000 lb. (450 kg.). The carbon electrode which was first used weighed about 1,400 lb. (635 kg.)

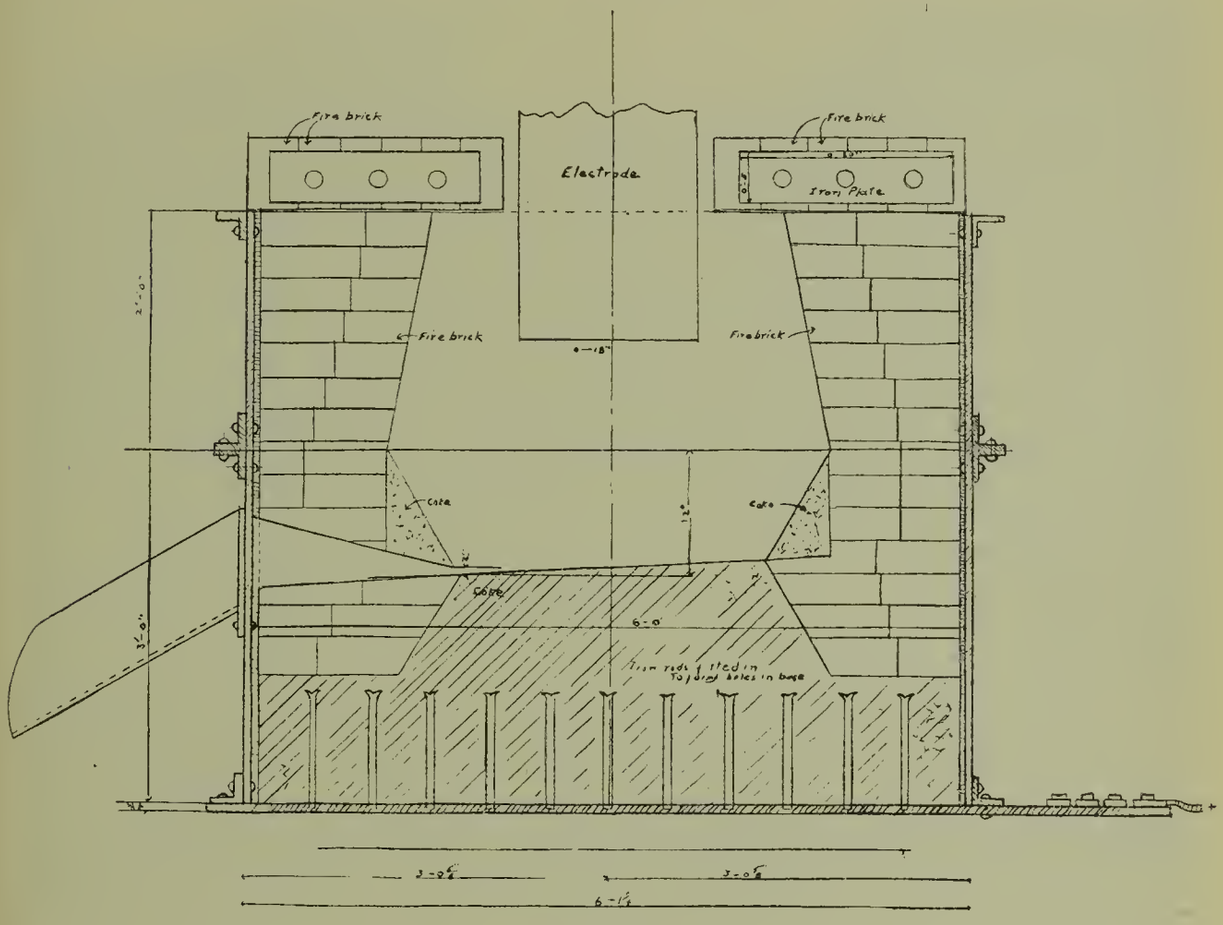


FIG. 8.

when new. During the operation of the furnace, if the current was shut of for more than an hour it would usually cause the furnace to freeze up. At first the coke caused trouble in the furnace, by accumulating, but this was eliminated by using finer coke, and a little less of it. We also noted a decrease in the electrode consumption when finer coke was used.

(*g*) *Electrode troubles.*—The electrodes which we first used were of carbon, built up from smaller electrode sections. The large electrode was 18 in. (45 cm.) square by 7 ft. (2.2 m.) long, made from 3 by 6 in. (7.5 x 15 cm.) carbon slabs. The first time

the electrode was heated by the arc it cracked and pieces broke off in several places. The pieces were picked out of the furnace, and ore with flux fed in, trusting that after a slag bath had been formed the cracking would stop, however this was not the case; after having to stop frequently to pick out solid pieces of the electrode weighing 25 to 50 lb. (11 to 23 kg.), the furnace was shut down, this electrode after so much damage being too short for further use. It was removed and the other carbon electrode placed in the holder.

Before striking an arc with the second carbon electrode a wood fire was placed beneath it in order to heat it up evenly. After two hours of this treatment, the electrode was placed in the furnace, contact made, and ore started in. The electrode, however, went to pieces.

I then ordered a copper bushing for the holder, so that we could use two Acheson graphite electrodes together. The Acheson electrodes were spliced so as to feed continually without waste. These electrodes held up finely and gave very good results. The dynamos gave us trouble, however, and we could only draw half load, consequently for nearly all the runs only one 8-in. (20 cm.) Acheson graphite electrode was used, which carried as high as 5,000 amperes at times.

(*h*) *Dynamo troubles.*—The dynamos are of the duplex, compound-wound type, made for this special work, but gave considerable trouble from sparking at the commutator brushes.

(*i*) *Ore trouble.*—The ore as it comes from the mine contains 40 to 50 percent water, as moisture. The ore is practically a mud. When I first went to Webster I had ore placed in the bins, and this had about six weeks in which to dry out. When I came to run this in the furnace it ran very nicely, but when it come to run the fresh ore from the mine, considerable difficulty was experienced. Sometimes part of the red hot charge would blow out in the men's faces and the air in the room was kept so full of dirt that it was almost uninhabitable. A dryer, of old steel plates, was rigged up and the ore partially dried in the sheds, using wood as a fuel.

(*j*) *Mining and tramming.*—The mining was done by open cut work. We built a small tram-road from one of the old cuts near

the smelter to the ore shed. The ore was hauled in a one-ton car, by a horse, from the cut to the ore bins. Ore from some of the other cuts was also used, this was brought in on ox carts. The higher grades of ore were picked, but for the bulk of the runs the ore as it came from the mine was used. On a large scale an aerial tramway would be the ideal way of handling the ore, as the deposit is located on a small hill, and deep mining

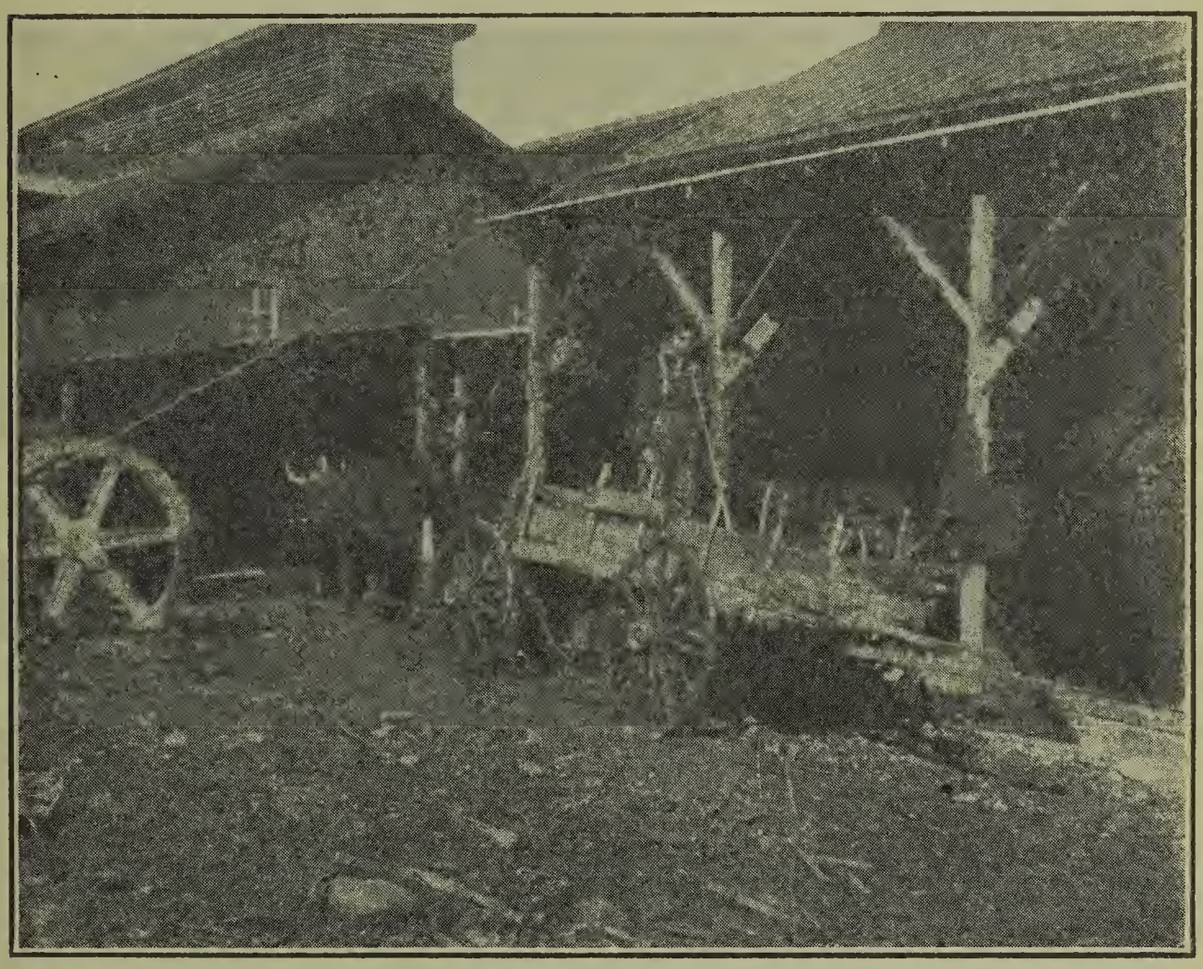


FIG. 9.

would not have to be resorted to for a long time. For the soft ores a steam shovel could be used. The hard ores are not difficult to break up, for they are easily drilled.

(*k*) *Efficiency of the furnace.*—The efficiency of the present furnace under the present conditions is not very high. The efficiency of this furnace as compared with one utilizing the waste gases to preheat and dry the ore, I would estimate at 60 to 70 percent. The fact that we used wet ore instead of dry ore makes a difference of 6 to 8 percent in the efficiency.

Furnace Runs.

Record of furnace run of Dec. 9, 1909.

CHARGES.

No. 1.	No. 2.
4 tons brown ore	3 tons brown ore.
1.02 tons limestone.	0.6 tons limestone.
0.48 tons coke.	0.48 tons coke.

Moisture in ore	42.50 percent
Total dry ore treated	8,050 pounds (3,660 kg.)
Net time of smelting	45.75 hours
Limestone used	3,600 pounds (1,635 kg.)
Coke used	1,940 pounds (882 kg.)
Power used, average kilowatts	170
Kilowatt hours used	7,755
Kilowatt-years used	0.896
Ore treated per kilowatt-year	9,000 pounds (4,090 kg.)
Ore treated per kilowatt-day	24.7 pounds (11.2 kg)
Nickel silicide obtained	1,242 pounds (565 kg.)
Percent of nickel in alloy	11.90
Silicide obtained per kilowatt-year	1,380 pounds (627 kg.)
Silicide obtained per kilowatt-day	3.8 pounds (1.7 kg.)

In other runs, from 8,250 lb. (3,750 kg.) to 10,160 lb. (4,620 kg.) of ore was treated, and from 975 lb. (443 kg.) to 2,230 lb. (1,014 kg.) of nickel silicide obtained per kilowatt-year of electric energy consumed. The power consumption is nearly constant per unit weight of ore treated, independent of its metal contents, showing that the chief worth of the furnace is the drying of the ore and the smelting of the slag. The silicide produced contained 10.50 to 22.60 percent of nickel.

The cost of production varies with the nickel content of the ore and of the silicide formed. The cost of smelting by hydro-electric power is only about 14 percent of what steam power cost us. The above figures are quite conservative in the case of the hydro-electric power.

(*l*) *Cost of operation.*—The cost of operation is based on the approximate cost as the plant was operated from October 1909 to April 1910, and goes to show the expense of operating or trying to operate in a primitive country. Two shifts of 11 and 13 hours respectively were run. We run the furnace about 4 to 5 days out of every week, very little repair work being necessary on the furnace. The engine gave us considerable trouble and during every run several stops were necessary for adjustment of the crank pin or the valves. The generators giving

the trouble they did, and forcing the plant to operate at 50 percent of full load, caused a great decrease in the efficiency of the power plant. The cost of smelting cannot therefore be reasonably figured from the cost of operation which I shall give. However in another part of the paper I will give the estimated probable cost of operation, using such data and figures as I have obtained from the present experiences.

Cost of operation per 24 hours:

1 Superintendent	$ 10.00
1 Assistant	2.50
2 Oilers @ $1.80	3.60
2 Firemen @ $2.40	4.80
2 Assistant Firemen @ $1.80	3.60
2 Head Furnacemen @ $2.50	5.00
4 Assistant Furnacemen @ $1.80	7.20
2 Miners @ $1.50	3.00
1 Blacksmith	2.00
1 Timekeeper	2.00
Coal, 18 tons, @ $4.50	81.00
Machine oil	4.00
Electrodes	9.10
Flux and coke	3.00
	$148.80

Our average power at the switchboard was 200 K. W. and we smelted on an average 7,800 lb. (3,500 kg.) of ore per 24 hours.

Cost of power:

$ 0.74 per kw.-day.
270.00 per kw.-yr.

Cost of power under full load would be:

$ 0.562 per kw.-day.
206.00 per kw.-yr.

In the above figures no account is taken of depreciation, interest, and repairs, which would undoubtedly raise the above figures 10 percent.

(*m*) *Output of plant.*—The output of the plant was limited to the available output of the generators, and varied from 800 to 1,200 lb. (365 to 545 kg.) silicide per day. This output on the present plant could be raised to 2,500 lb. (1,135 kg.) of nickel silicide, by operating the generators at full load, and using a dryer and preheater with the furnace. However, the furnace has served its purpose for a commercial demonstration, and has

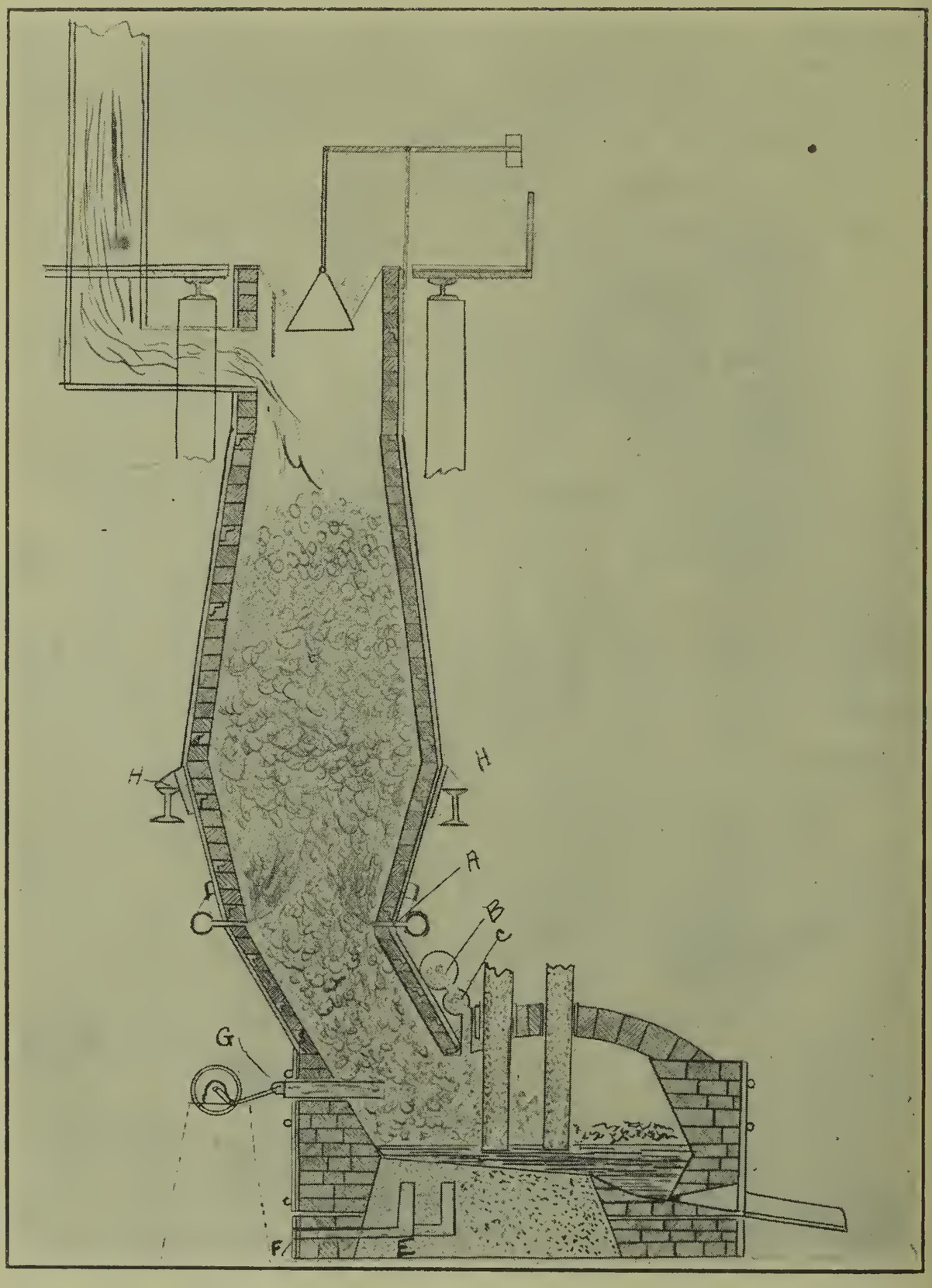


FIG. 10.

given the operators a chance for observations which are only possible with a furnace of this kind. I have suggested several improvements, and have sketched some of my ideas on paper. I will discuss some of the merits, advantages, and disadvantages of the furnace designs. In the furnace designs I have included several features which have been patented by Doctor Heroult and others, but as these furnaces are not to be constructed as yet, there is no harm in giving them here.

VII.

CONCLUSIONS.

(*a*) *The shaft furnace.*—The shaft furnace for electric smelting is the ideal furnace, however, since the flow of gases is slow compared to the air blast furnace, it does not need to be so very high. For ordinary operations on the mixed nickel ores a shaft furnace should not be more than 10 to 12 feet high, *i. e.*, above the fusion zone. There is considerable fines in the ore, which would cause trouble with a higher shaft. For this ore a dryer would be necessary, utilizing the waste gases from the furnace. Figs. 10 and 11 give a general idea of the shaft furnace I have in mind. The ore with the flux is introduced at the top of the furnace from the charging floor. The furnace has a fore hearth for the collecting of slag and metal. The furnace is designed to carry ten 8-in. (20 cm.) graphite electrodes. The ore enters the crucible back of the electrodes. To aid in the distribution of the sticky ore mass, a set of plungers (G) are arranged at back of furnace. The furnace is designed for direct or alternating current (single phase). The bed of the furnace acts as one conductor, the dotted portion shows this bed. Tubular conductors carry the current to iron distributers in the bed of furnace, which is heat insulated. Coke is introduced at (c) independent of the charge; by so doing the electrode consumption is lessened; air enters for combustion of gases in the shaft at (A). I have found that, by introducing the coke or reducer around or near the electrode, the consumption of electrode can be cut down one-half. Each electrode is operated automatically and independent of the others. Although the shaft furnace is very good in theory, I do not think it practical at this time for the ore in question. The

ore is extremely refractory and shut downs are frequent. The cost of shutting down a shaft furnace and repairing would be very high, therefore for this and other reasons I think a shaft furnace

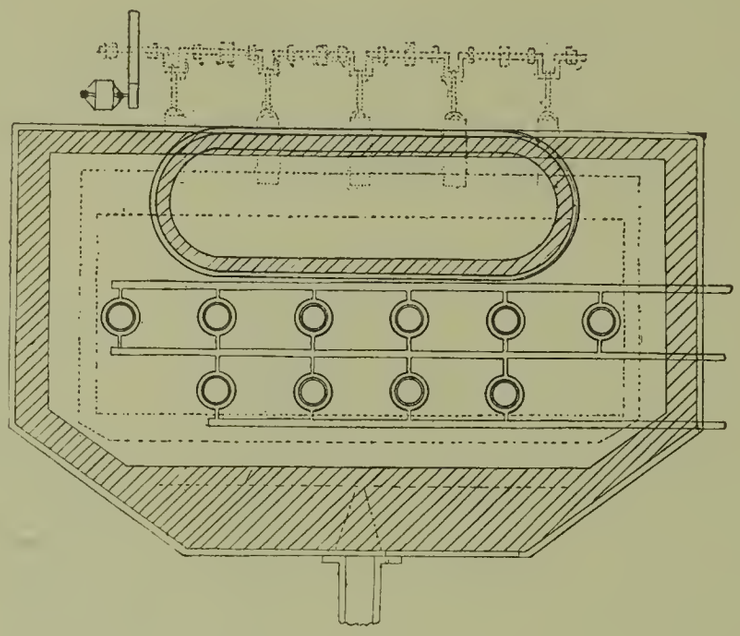


FIG. 11.

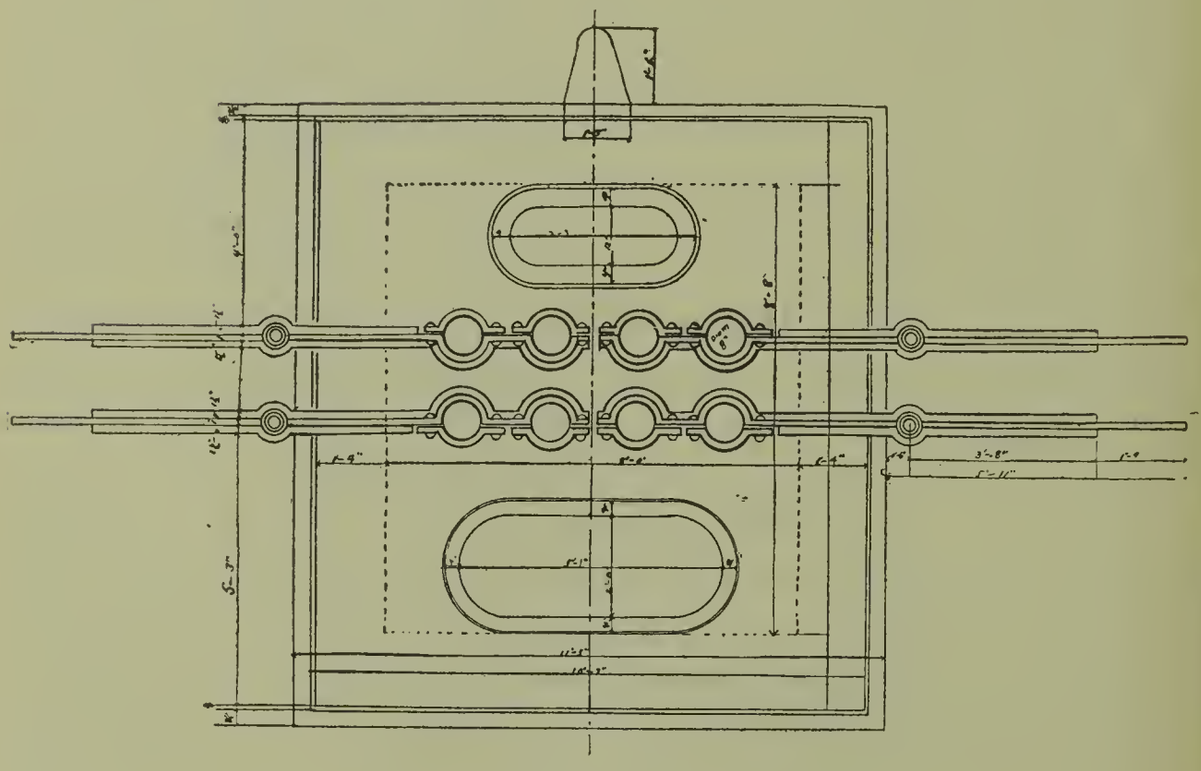


FIG. 12.

and preheater would work out better. If it were desired to run the extremely fine ore alone at any time, then the crucible furnace would be the one to use. It would be impossible to run

the extremely fine ore in a shaft furnace successfully. In the treatment of iron ores and other ores, the shaft furnace no doubt would be all right.

(*b*) *The crucible furnace.*—By crucible furnace I have reference to a furnace which is comparatively shallow and has no shaft for preheating and reducing the ores.

The crucible furnace illustrated in Figs. 12 and 13 is of a special design, which I sketched, after careful observations on the present furnace. The furnace as shown is designed for direct current, which at the present time is installed. The furnace is designed to carry eight 8-in. (20 cm.) Acheson graphite electrodes, operating under a maximum load of 2,000 electric h. p. The electrodes are worked in pairs and controlled by automatic hydraulic regulating device. The electrodes are carried in cast steel holders, to which are fastened eight flexible cables. The holders are supported on a three-inch steel rod, which works in a hydraulic ram Fig. 13. To give the rod a free movement a special guide was designed, shown in Fig. 14. A is a top view and B a section thru MN. The ball bearings will not allow the rod to hang if properly lubricated and kept covered. Fig. 13 gives a front view of the furnace, showing position of spout and electrodes. Fig. 10 is a side view of the furnace and gives an idea as to method of making the lower contact. The conductors are heavy copper tubing which enter the lower bed of the furnace, to these are secured wrought iron distributers. The carbon bottom of furnace is not brought to the outside wall of furnace but to the brick lining, which cuts down the radiation losses. There are openings in the top of furnace for electrodes, and a flue for escaping gases and an opening for ore. The gases are taken off from the front part of the furnace and burned in the preheater. The ore as it leaves the preheater is dumped into the furnace at the rear. The two flues are brick lined. The interior of the furnace is lined very much in the way the experimental furnace was lined. The electrodes are water jacketed, below the water jacket is a jacket constructed for the introduction of coke around the electrode. In Fig. 15 is a plan of jacket, B and C are sections. W shows section of water jacket. The water enters at E and flows out at R. K represents section of coke jacket. The coke enters at M and is fed by a screw device.

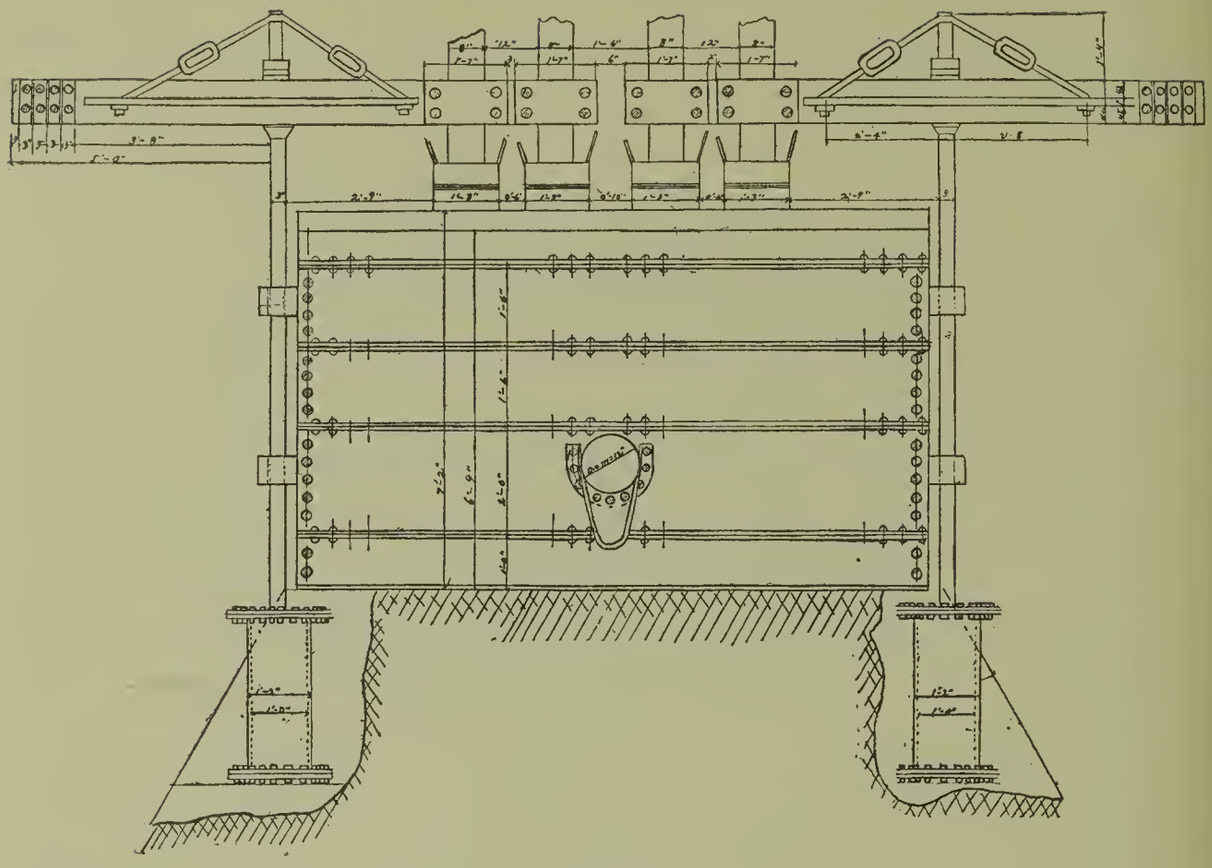


FIG. 13.

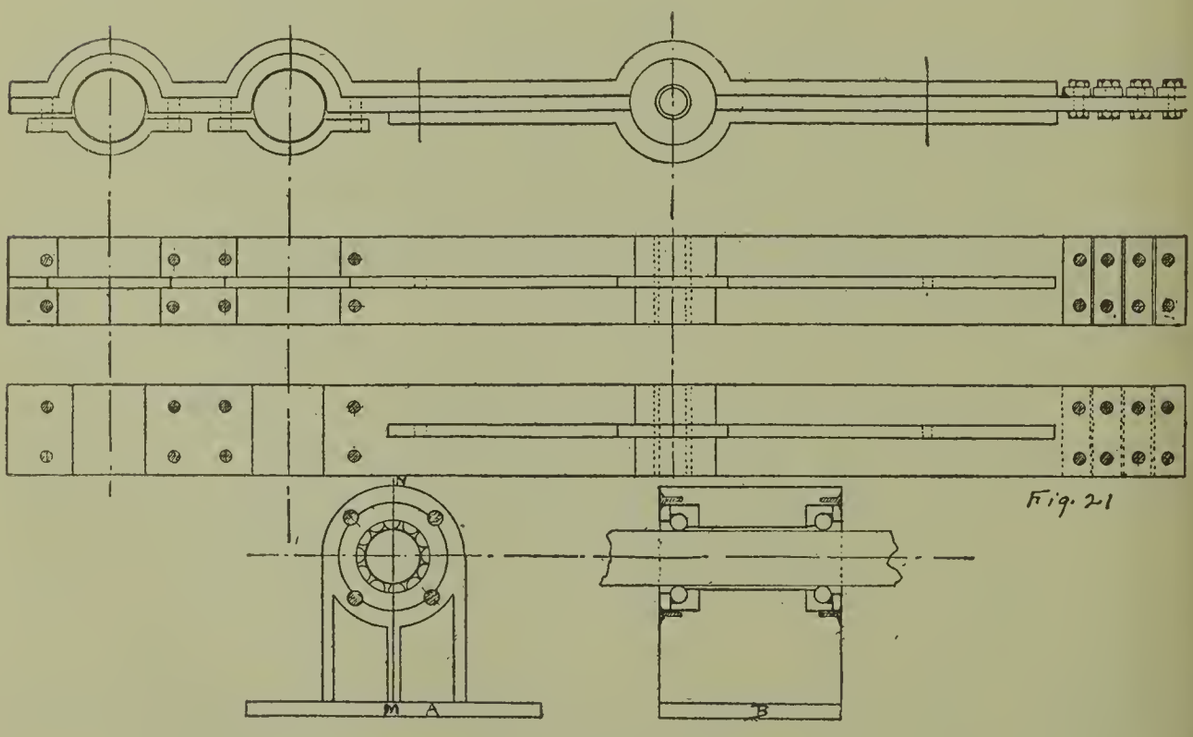


FIG. 14.

General plan of introducing coke to electrodes is shown in Fig. 16, feeding coke to four electrodes, the other four being taken care of in the same way. The two jackets are suspended on tie rods, then built in top of furnace. They are built in two sections so they may be easily renewed. For the first operations it is intended to feed the coke intermittently by hand feed. By introducing the coke at this point, an extremely high reducing atmosphere is constantly kept around the electrode, consequently the electrode consumption is cut down. As to the reducing power, or action on the ore by this method, it is about the same. The fusion zone is limited to a few inches immediately around the tip of the electrode, extending perhaps 8 or 10 inches (20 to 25 cm.). Under normal conditions there is a violent bubbling and boiling, which causes the fine coke 10 to 20 mesh (2.5 to 5 mm.) to intimately mix with the fused mass and bring about reduction. On a small scale I have found that this method of introducing the coke works out very nicely. With frequent tappings the excess of coke is carried away in slag.

(*c*) *Electric smelter design.*—Figs. 17, 18, show an electric smelter design for the treatment of this ore. It consists of the generator room, furnace room, and ore bins. The generator room contains the motor-generators for the changing of the current from three phase to single phase. The room contains two 3,000 kw. motor-generators, the exciter, the high-tension switchboards, transformers and recording-meters. Current is to be furnished by outside power stations. In the furnace room, we have the furnaces, dryers, switchboards, slag and metal conveyors, hydraulic accumulator, and pumps. It is intended to keep three furnaces in operation, and have one for emergencies, which can be used at any time. All electric cables are carried in conduits, except the cables to the electrode holders. The hydraulic control is so arranged with each furnace that it may be regulated by hand, or automatically. All metal and slag is tapped into an iron trough of water, and is carried to the concentrating room by endless conveyor, where slag is flushed to dump. The dryer is of such length that the ore is dried and preheated before entering the furnace. A check draft and explosion door is arranged at end of dryer. Each dryer has a stack to give better regulation.

The limestone and ore are crushed before being placed in the

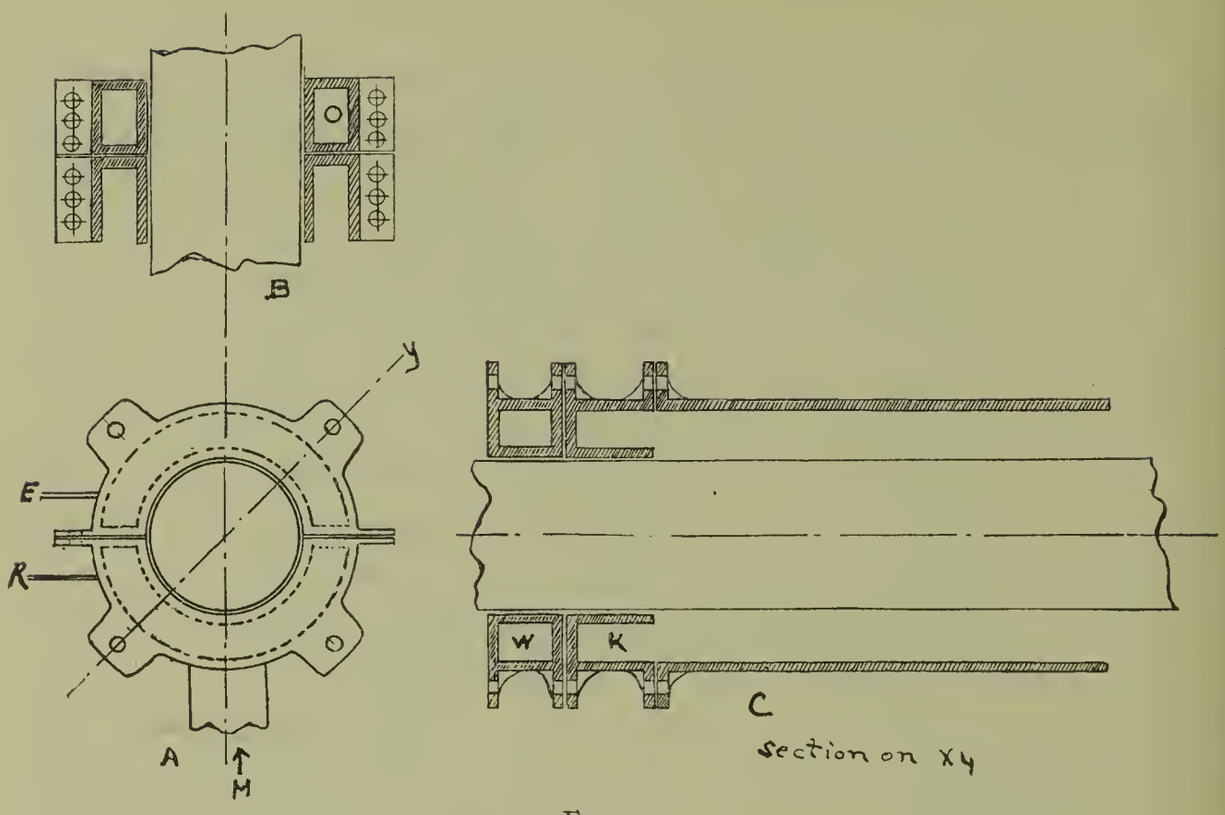

B
y
E
R
A
M
W
K
C
Section on xy

FIG. 15.

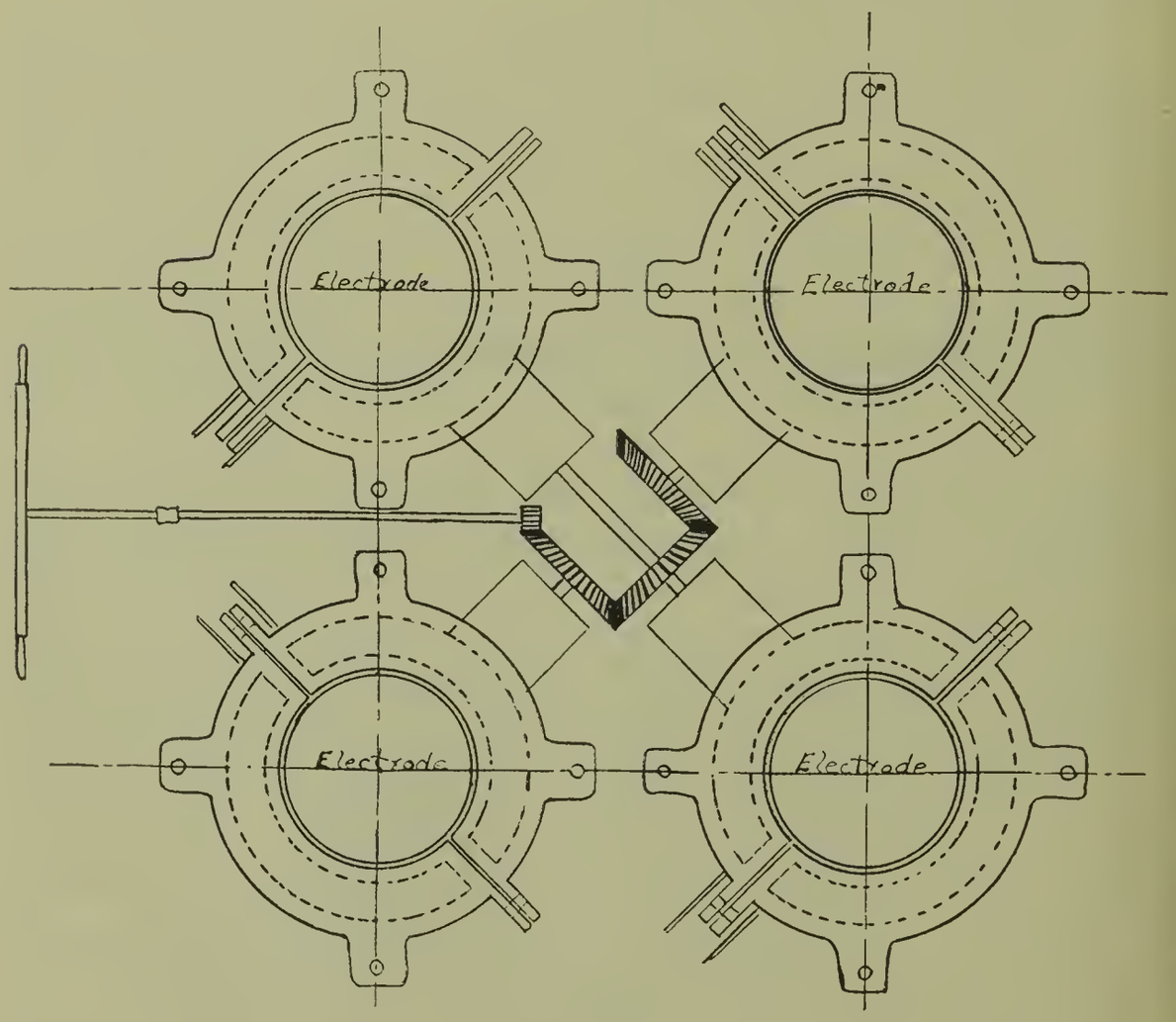

Electrode
Electrode
Electrode
Electrode

FIG. 16.

bins. From the bins they are drawn into weigh cars, then dumped into the feeding hopper of the dryer. A screw feed regulates the amount of ore being fed into the dryer.

(*d*) *Cost of 100-ton plant.*—Approximate cost of a 100-ton electric smelter, including aerial tramway, rock house, crusher, cars, buckets, etc.:

Ore bins	$ 3,000
Buildings	12,240
Tramways, cars and equipment	25,000
Weigh car	1,000
Crusher	900
Pulverizer for coke	800
Motors	1,600
Furnaces (5)	44,000
Motor-generators	45,000
Transformers	20,000
Structural work	22,000
Dryers and motors	15,000
Switchboard and equipment	30,000
Erection, labor, materials, etc	60,000
Total	$280,540

Although these figures appear quite high, from past experience I think them conservative.

(*e*) *Cost of operation.*—Estimate as to cost of operation of a 100-ton smelter, figuring operating 300 days per year, and cost of power $25.00 per kw.-yr.

Expense per day:

Interest and depreciation (14%)	$130.00
Mining @ 15 cents per ton	15.00
Power	450.00
Labor and supervising	75.00
Electrodes	130.00
Repairs	10.00
Coke and limestone	75.00
Total cost	$885.00

From 100 tons of ore we would extract on an average 15 tons of ferro-nickel-silicide.

$885.00/15 = $59.00 per ton, average cost of metal alloy.

(*f*) *General remarks.*—The present plant for commercial oper-

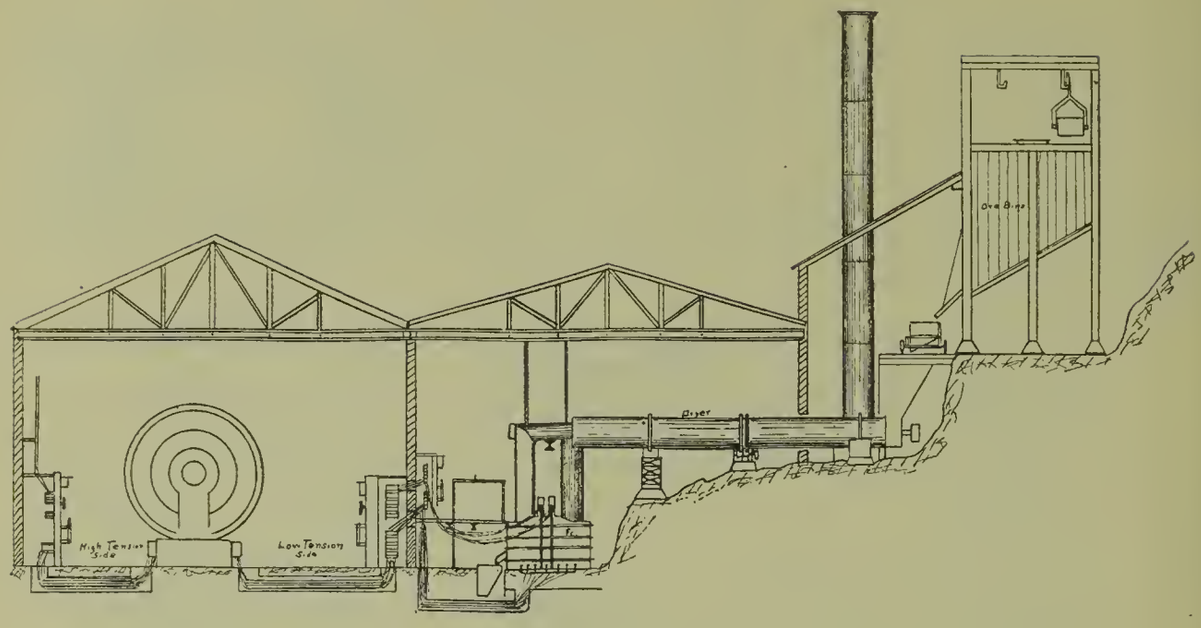

Ore Bins
River
High Tension Side
Low Tension Side

FIG. 17.

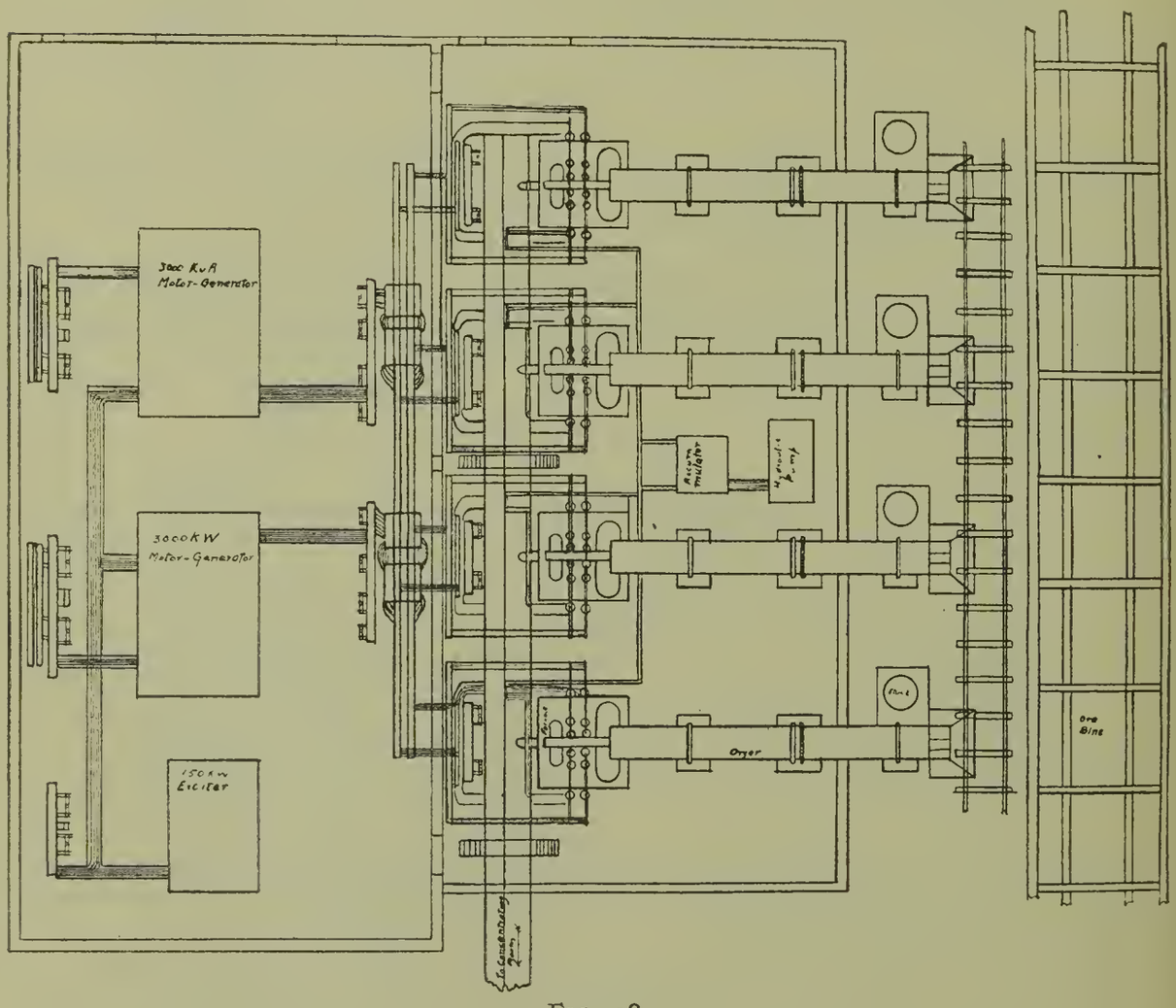

300 K.V.A Motor-Generator
3000 KW Motor-Generator
150 K.W Exciter
Auxiliary Pump
Hydraulic Pump
Ore Bins

FIG. 18.

ations is out of the question. The wet process now existing is also out of the question. The only process available for this ore at present is the electric furnace process in a first-class, efficient plant.

There is no doubt in my mind but that the present cost of producing the alloy can be materially lessened by further investigations and improvements.

A paper read in abstract by E. F. Roeber, at the Twentieth General Meeting of the American Electrochemical Society, in Toronto, Canada, September 21, 1911; President W. R. Whitney in the Chair

THE DIRECT PRODUCTION OF MOLYBDENUM STEEL IN THE ELECTRIC FURNACE.

By E. J. Dittus and R. G. Bowman.

(Extracts from a Thesis presented to the Colorado School of Mines.)

Preface.

It is the intention of the authors to present in the following paper a brief résumé of the properties, uses and methods of production of molydenum steel, and to describe in detail a series of experiments on the production of molybdenum steel, direct from iron ore and molybdenite, in the electric furnace.

The use of a sulphide ore of so active a metal as molybdenum in connection with the manufacture of steel presents a number of problems. Foremost among these are the complete reduction of the molybdenum without serious loss, the diffusion of the molybdenum through the steel to form a homogeneous product, and the elimination of the sulphur from the steel. The process employed was one based on a reaction described by F. M. Becket in U. S. Patent 855157, and is believed to be new.

The authors wish to express their hearty appreciation and thanks to Professor W. G. Haldane and others for the encouragement and assistance they have given in carrying out the experimental investigations.

The desirable properties of molydenum steel and the expense involved in its manufacture by the methods at present in use, led the authors to search for some reaction upon which could be based a process for its direct production from its most common ore. Without such a process any extended use of molybdenum steel, instead of tungsten steel, is very improbable. The reaction described by Becket appeared the most simple and was, therefore, made the subject of the investigations.

The production of steel direct from ore in the electric furnace is easily accomplished where the ore is pure. Certain impurities,

particularly sulphur, are, however, very difficult to remove from the metal. Small amounts may be removed by the use of a basic slag but this method is limited in its application since the fusibility of the slag decreased rapidly with increasing basicity. For the reduction of molybdenite in the presence of molten iron it is necessary to have present some substance which has a greater affinity for sulphur than either molybdenum or iron. The compound formed by the desulphurizing agent and the sulphur must either pass into the slag or be volatilized as soon as formed. The two metals which seem best adapted for use as desulphurizers, under these conditions, are manganese and silicon.

Manganese in the form of metal or as the ferro alloy reacts with sulphides at high temperatures according to the following reaction:

$$2\,Mn + RS = R + 2MnS.$$

The manganese sulphide forms a slag resembling iron sulphide. If the above reaction takes place in a bath of molten steel or iron the resulting metal is apt to contain small included masses of manganese sulphide. Sulphur in this form has little effect on the properties of steel. This reaction might be applied to the production of alloy steels, such as molybdenum steel, by adding a mixture of molybdenite and ferro-manganese to the bath of molten steel just before tapping. This would result in the formation of ferro-molybdenum and manganese sulphide; the former alloying with the steel and the latter passing into the slag.

Silicon reacts with sulphides to form silicon sulphide SiS_2 and liberates the metal of the original sulphide. This is the reaction described by Becket.[1] The reaction has been investigated by Fielding[2] who produced a yellow powder which sublimed at 1,500°C and which decomposed water with the formation of H_2S and silicic acid. This compound did not correspond to the formula SiS_2. Sabatier[3] describes a somewhat similar compound and suggests the formula Si_3S_4. The heat of formation of SiS_2 is given by Sabatier[3] as + 40.4.

This reaction might be applied to the production of molybdenum steel in the same manner as the manganese reaction

[1] Elec. Chem. & Met. Industry, Aug., 1909.
[2] Bul. de Soc. Chem., Paris **2**, 38, 153.
[3] Comptes Rendus, **90**, 819.

described above. The silicon, in the form of ferro-silicon, and the molybdenite should be reduced to powder, intimately mixed in the proper proportion to produce the reaction and added to the steel in the furnace before tapping. The mixture might be added in a soft iron tube or in small briquettes made up with a binder of sodium silicate. Addition in the ladle during tapping would propably result in raising the sulphur content of the metal on account of the absence of slag, which in the furnace would remove small amounts of sulphur which might tend to pass into the steel.

Experimental Work.

The experimental work of this thesis was undertaken with a view to determine the feasibility of applying the Becket process to the production of molydenum steel direct from iron ore and molybdenite in the electric furnace. The problems presented were: first, the design and construction of a suitable furnace; second, the production of a carbon steel by direct smelting from iron ore; third, the addition of molybdenum to the steel and the elimination of the sulphur of the molybdenite from the bath of metal. Of these the third presents by far the greatest difficulty.

Furnace.

To accomplish the results desired it was necessary that the furnace fulfill the following requirements: the crucible walls should be of a neutral or basic material, free from carbon; the furnace should be easily charged and operated; the electrodes should be capable of a variety of adjustments; the furnace should be capable of being readily dismantled for the purpose of relining or making alterations. Since the best method of heating could be determined only by experiment, it was thought best to design a furnace capable of being operated as a Girod, or with slight alteration, as a Heroult furnace.

With the above mentioned requirements in view, two furnaces were designed. These were essentially the same in principle but differed in the proportions of the crucible and in certain minor details. A detailed description of the design illustrated in Fig. 1 will suffice, since this was the furnace finally adopted and used in the investigations.

The crucible is elliptical in cross section and measures 6 in. x

9 in. x 8 in. (15 x 23 x 20 cm.) deep. The total volume of the crucible is about 360 cu. in. (5.5 litres), the volume of the smelting zone is about 90 cu. in. (1.4 litres). The walls have a slight bosh to effect a concentration of heat at the base of the crucible. The crucible lining is of burned magnesite obtained by crushing magnesite brick to pass 10 mesh (2.5 mm.). The magnesite was mixed with tar to form a paste and rammed in hot around a central wooden form. The tar was burned out in the heating up of the furnace.

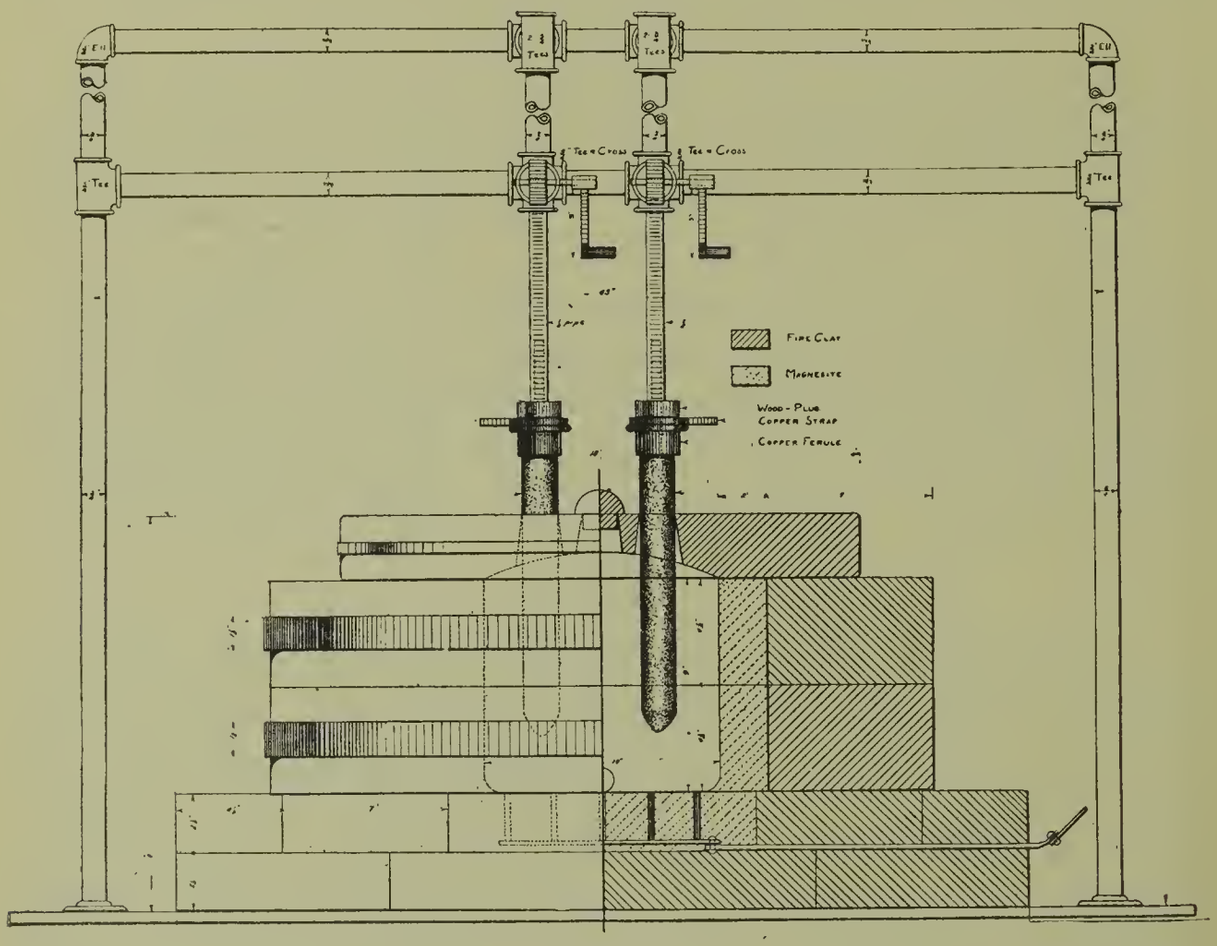


FIG. 1.

The walls are built up of eight fire-brick sections of special design, luted together with fire-clay and encircled by steel bands. These rest on a foundation made up of two courses of standard fire-brick, with the joints filled with fire-clay.

The cover for the crucible is a solid elliptical fire-brick section 17 in. x 20 in. and 2½ in. (43 x 50 x 53 cm.) thick, encircled by a steel band. The electrodes enter through the slot in the center of the cover. This slot admits of considerable lateral or angular movement of the electrodes. The under side of the cover is

slightly concave. An asbestos gasket is placed under the cover where it rests on the top of the furnace, and small asbestos washers encircle the electrodes where they pass through the cover.

The tap-hole is 1¼ in. (3 cm.) in diameter, and is well rounded at its point of entrance to the crucible. A small hole 1 in. (2.5 cm.) in diameter extends diagonally from the top of the outer wall to the smelting zone for the purpose of pyrometer readings and for the escape of gases.

Electrodes.

The hearth electrode consists of six ½-in. (1.3 cm.) rods of Swedish iron screwed into a ⅜-in. (0.9 cm.) plate of the same material. The plate is embedded in the lining of the bottom of the crucible in such a position that the upper ends of the rods are flush with the inner surface. A copper strap 3/16 in. (0.5 cm.) thick and 1.5 in. (4 cm.) wide, bolted to the plate, extends outside the furnace for connection to the source of current.

The two movable electrodes are supported on a frame above the furnace. The supporting frame is entirely independent of the furnace proper and is therefore unaffected by expansion or contraction of the walls. The electrodes are cylindrical graphite rods 1.25 in. (3 cm.) in diameter and 11 in. (28 cm.) long. Each electrode is attached to a brass rod by means of a collar clamp and cotter pin. The rod carries a rack which meshes with a pinion mounted on the electrode support. The electrode supports are mounted to slide from right to left on the supporting frame. This facilitates adjustment of the electrodes while the furnace is in operation, and also allows the electrodes to be pushed back out of the way when relining the crucible. The support is so constructed that the electrode may be swung about it as a center and inclined at any angle. Ordinary pipe fittings were used as much as possible in the construction of the frame and supports, since these give a light, strong construction, and require no special castings or forgings. An electrode support built entirely of pipe and fittings is illustrated in Fig. 2.

The furnace illustrated in Figs. 2 and 3 differs from that described above in having a crucible of double the depth de-

scribed, *i. e.*, 16 in. (40 cm.), and a different arrangement of the rods in the bottom of the crucible. This furnace was designed to operate continuously with a column of cold charge resting on the molten material, as in a shaft furnace.

This type of furnace may be operated as a Girod steel furnace by connecting the two movable electrodes in parallel with one side of the circuit and the hearth electrode with the opposite

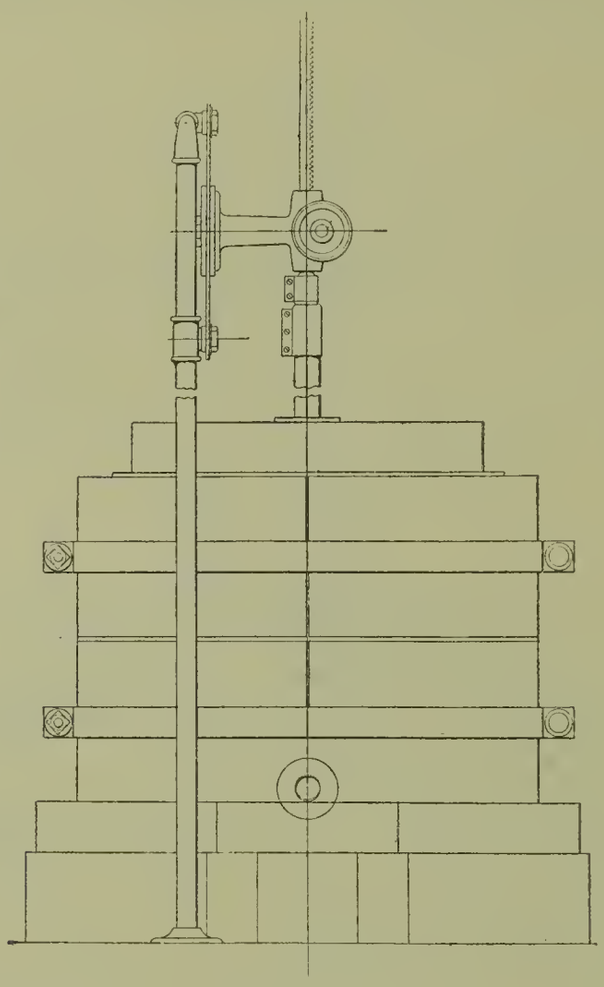


FIG. 2.

side. The movable electrodes may then be placed parallel vertically, thus forming two arcs, or set to converge to a single central point forming but one arc. To operate the furnace as a Heroult steel furnace, the points of the rods in the hearth electrode are covered with a layer of magnesite, and the two movable electrodes are connected in series with the circuit. The magnesite covering for the hearth electrode need not be of any great thickness, since there is but a slight tendency for the current to cross through the iron plate after the charge becomes molten.

Running of the Furnace.

The furnace is charged at the beginning of a run by removing the cover and distributing the charge with a trough or funnel,

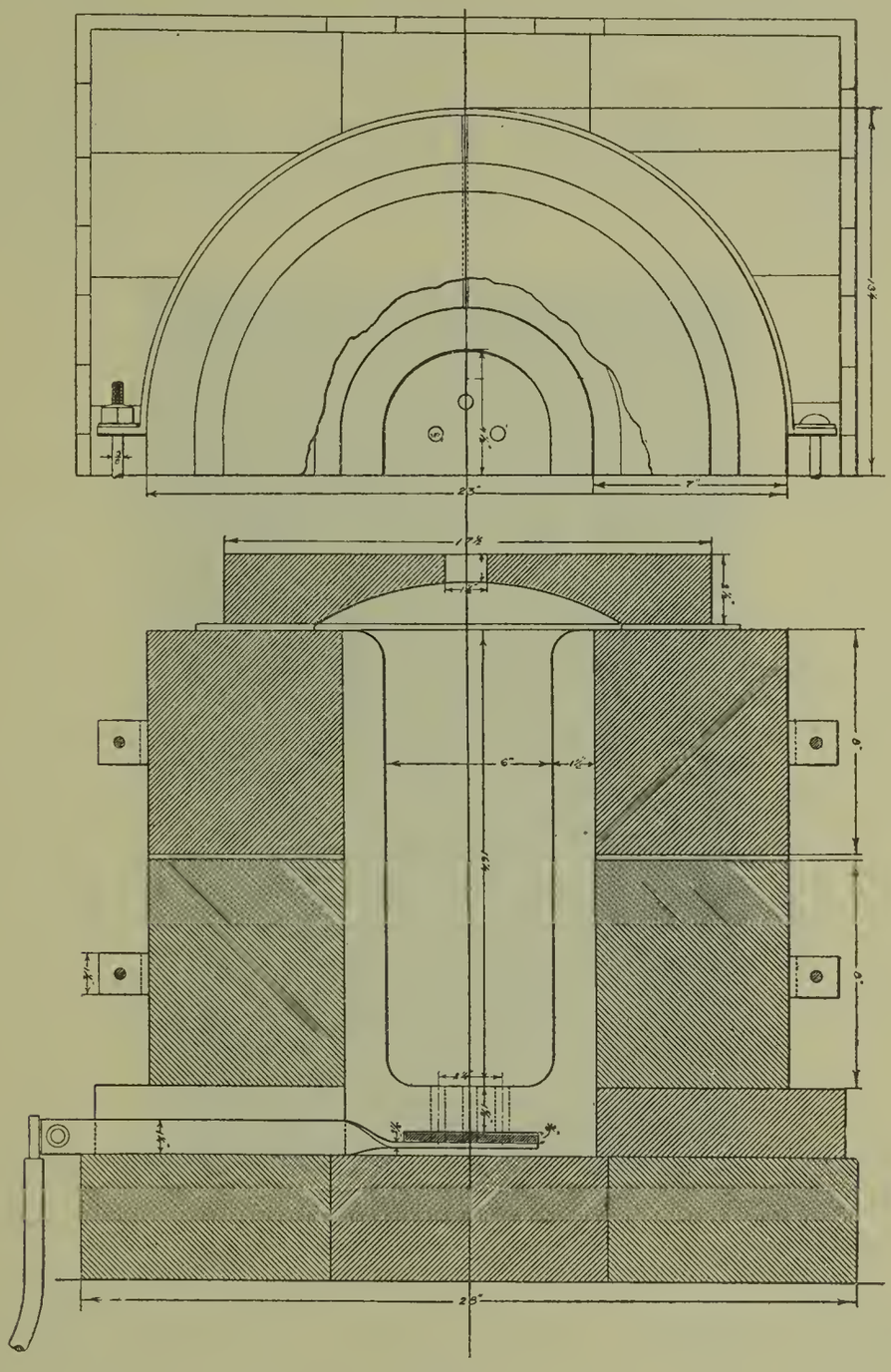


Fig. 3.

Additional material may be added through the opening in the cover while the furnace is running.

The small amount of coke in the charge used was found to be sufficient to make the cold charge slightly conducting, the resistance rapidly decreases with rise of temperature, and the furnace may thus be readily started on a cold charge, providing

the walls of the crucible are hot. The electrodes are forced down through the charge to within a short distance of the bottom of the crucible at starting. As the charge becomes hot, the electrodes are gradually raised until the charge is reduced and the arcs play between the ends of the electrodes and the surface of the slag. With the current available it was found impossible to maintain arcs below both electrodes simultaneously for any length of time. The best results were obtained by raising and

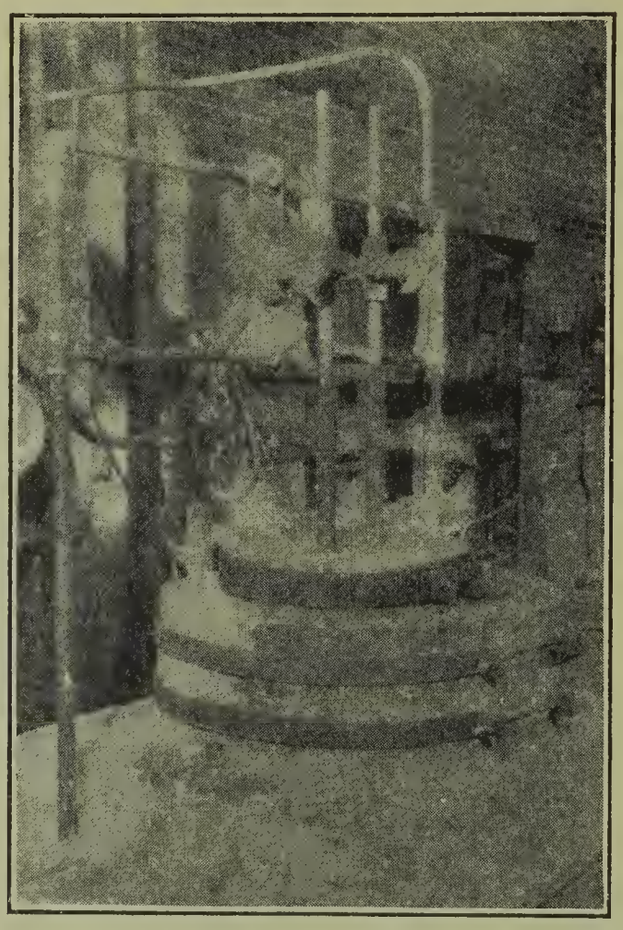


FIG. 4.

lowering the electrodes alternately at intervals of two or three minutes, which caused the arcs to alternate from one electrode to the other.

The capacity of the furnace as described is about 1,000 gm. of metal per charge, or approximately 2,000 gm. of raw charge. A charge of 1,000 gm. was employed in several runs. This yielded 500 gm. of metal, but this amount is too small to make clean tapping possible.

Single-phase alternating current at 60 cycles, 25 volts, was

employed. The amperage varied throughout the run, but averaged 250 with the arcs running steadily.

An opening for pyrometer readings was provided in the furnace, but no readings were taken. In a furnace of this size a variation of temperature from cold ore to the maximum smelting heat may be found in a space of a few inches; such a variation would make pyrometer readings irregular and of little value.

The generator used in supplying current for the experiment was a General Electric alternating current composite-wound, having both slip rings and a rectifier commuter for supplying

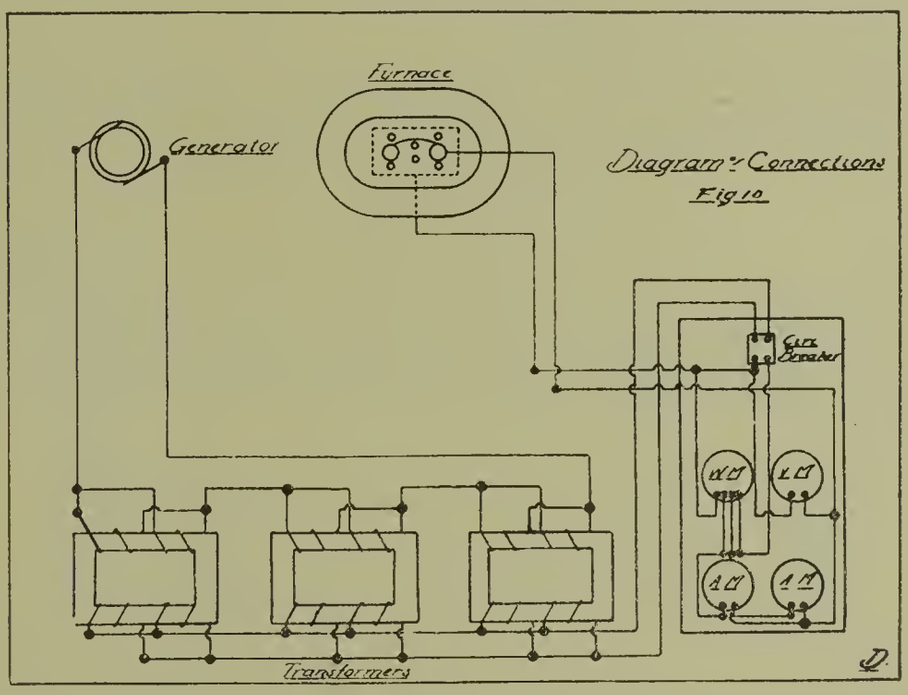


FIG. 5.

direct current to the series field. The name plate gives the following data: No. 2044, Type A, Class 10-30—1500. No load voltage 1040, Full load voltage 1155, Thompson-Houston System.

The transformers were made by the General Electric Co. No. 423052, Type H, Cycles 60, Form G, Volts 1,200, 2,400, 120, 240. The transformation ratio was 10-1. The primaries were connected in series with the line and the two coils in parallel with each other, the secondaries all in parallel with the line. This combination gave the best results. The amperage ranged from 0-500 with an average of 250, the voltage averaged 25.

The switchboard consisted of two ammeters in parallel with each other and in series with the line, a voltmeter in parallel, a

circuit breaker, and recording wattmeter. The ammeters were made by Westinghouse Electric Co. 7,200 alt. circuit, cap. 300 amps. The voltmeter was a Thompson Portable machine, the circuit breaker an I. T. E. type W. 500 amps. The wattmeter was a Thompson recording, No. 104114, Type M, Class 100-200 Form D_3.

During all the work the generator was run with the series coil short circuited across the brushes. This protected the machine because any overload or short would cause the voltage to drop due to the armature reaction and reactance. A dead short would thus cause the voltage to drop to zero and thus no harm could be done to the machine.

The connections of the various pieces of apparatus are illustrated in the accompanying diagram, Fig. 5.

Ores and Fluxes.

The iron ore employed was a good grade of hematite from the mines of the Colorado Fuel and Iron Co. at Sunrise, Wyoming. Two separate lots were used, the analyses of which were as follows:

Fe	66.06	percent
Al_2O_3	1.52	"
SiO_2	4.12	"
P	0.04	"
S	0.04	"

The ore was crushed to pass eight mesh (3 mm.) and the fines were retained.

Molybdenite.

A quantity of very pure molybdenite from Yorkés Peninsula, Australia, was obtained from the Foote Mineral Co.

Analysis showed the material to be practically pure MoS_2 with only a trace of impurity in the form of copper sulphide.

Ferro Silicon.

One lot of ferro-silicon was used, the analysis of which was as follows:

Fe	49.70	percent
Si	50.00	"
S	0.005	"
C	0.20	"

The ferro silicon and molybdenite were ground to pass thirty mesh (0.85 mm.) and intimately mixed in the proper proportions to bring about the reaction:

$$MoS_2 + Si = Mo + SiS_2.$$

The proper amount of this mixture to give the desired molybdenum content in the steel was enclosed in a small paper tube and added to the bath of metal just before reduction was complete.

Coke.

The coke used was from Trinidad, Colorado, and was obtained from the North American Smelting Co., of Golden, Colorado, from whom the analysis was also obtained.

Analysis:

Fixed carbon	77.70	percent
Vol. and combust. mat.	2.80	"
Ash	18.66	"
Fe_2O	1.23	"
Al_2O_3	1.41	"
CaO	1.30	"
SiO_2	6.59	"
S	0.47	"
Moisture	0.84	"

The coke was crushed to pass eight mesh (3 mm.) and the fines retained.

Lime.

Ordinary builders' lime was used as flux. The analysis was as follows:

CaO	92.76	percent
MgO	——	
Fe_2O_3, Al_2O_3	1.90	"
SiO_2	1.34	"
P	0.06	"

The lime was crushed to pass eight mesh (3 mm.) and the fines retained.

Record of Furnace Results.

Run No. 5.

Charge:

Iron ore	3,025 gm.
Coke	660 "
Lime	412 "
Calculated iron content	2,000 "
Length of run	1 hr. 30 min.
Mean amperes	200
Mean volts	25
K. W.	5
Metal obtained	1,700 gm.
Calculated molybdenum content	2.5 percent

Iron turnings were spread over the bottom of the crucible and covered by a layer of coke, lime and silica to form slag and protect the iron. The metal melted in 25 minutes but was kept molten in the furnace for 15 minutes after fusion in order to burn out the tar from new portions of the lining. This metal was tapped cleanly and a charge sufficient for 500 gm. of metal was added. This reduced in 20 minutes. Molybdenite-ferro-silicon mixture sufficient to give a content of 5 percent Mo. in the metal was added in a paper tube just before reduction was complete. The tap hole was opened but the metal was level with the tap and only slag flowed out. The tap hole was again plugged and a charge sufficient to give 1,000 gm. of metal was charged.

Reduction was rapid and the charge was entirely reduced in 30 minutes. Molybdenite mixture sufficient to give a molybdenum content of 2.5 percent in the metal was added just before final reduction of the charge. Since the bottom of the crucible appeared to be sinking constantly, another charge equivalent to 500 gm. of metal was added and reduced in 15 minutes. This gave a total of 2,000 gm. of metal (calculated) in the furnace, with sufficient molybdenum to yield 2.5 percent in the total.

The tap hole was opened and slag flowed out freely but no metal was obtained. The current was shut off and the furnace allowed to cool. A solid mass of metal level with the tap hole was found in the bottom of the crucible.

The furnace was taken down and the entire lining and bottom electrode removed. A solid mass of metal weighing 1,700 gm. was obtained. The metal was malleable and tough, and was broken with great difficulty. When broken across the center the

fracture was very fine grained and dense at the center of the mass and coarsely crystalline and full of small blow holes at its outer edges.

A section of the central portion of the mass, polished and etched with picric acid showed a network of a bright white constituent, probably a double carbide of molybdenum and iron. A section in the coarsely crystalline portion of the mass showed a coarsely granular structure.

Analysis:

C	0.62	percent
Si	0.91	"
Mo	1.15	"
P	0.08	"
S	0.37	"

Run No. 6.

Charge:

Iron ore	756	gm.
Coke	163	"
Lime	103	"
Calculated iron content	500	"
Calculated molybdenum content	2	percent
Length of run	30	min.
Mean amperes	300	
Mean volts	25	
K. W.	7.5	
Metal tapped	—	

Description of run:

The furnace was rebuilt and arranged as in Run 5. The tap hole was given a slightly greater inclination.

Iron turnings were placed on the bottom of the crucible and were melted in 30 minutes. The tar in the new lining burned out very slowly and the molten metal was therefore allowed to remain in the furnace for one and one-half hours with the arcs running. When the greater part of the tar was burned out the metal was tapped and the charge added.

Reduction was rapid and molybdenum mixture sufficient to give a molybdenum content of 2 percent was added. The walls of the crucible were crumbling and the crucible was rapidly becoming too large to heat properly. The magnesite from the lining made the fused charge thick and pasty. An attempt was made to tap the metal but nothing was obtained. The crucible was now too

large to work properly but since the bottom seemed to be sinking it was thought advisable to fill the crucible up to the proper level by the reduction of a large charge of ore. A charge corresponding to 2,000 gm. of metal was added in small amounts with the arcs running. Reduction was rapid but not uniform owing to the increased size of the crucible. By continual poking of the charge the whole mass was finally fused but was not sufficiently liquid to tap. The furnace was allowed to cool and a mass of metal level with the tap hole was found embedded firmly in the bottom. A small portion of this metal was removed with tongs and proved to be very soft and malleable and easily sawn or cut.

A polished section etched with picric acid showed a network of bright white lines in a ground of pearlite.

Analysis:

C	1.14	percent
Si	1.23	"
P	0.03	"
S	0.03	"
Mo	0.45	"

Run No. 7.

Charge:

Iron ore	1,512	gm.
Coke	326	"
Lime	206	"
Calculated iron content	1,000	"
Length of run	60	min.
Mean amperes	300	
Mean volts	25	
K. W.	7.5	
Metal tapped	—	

The crucible was relined. The electrodes were partially consumed and, since no more of the same size were at hand, were removed and replaced by 1⅛-in. (3 cm.) rods. The arcs were started on the mass of metal remaining from Run 6. When the furnace was hot and this metal was molten, a charge was added and quickly reduced. The tap-hole was opened, but only a portion of the metal was obtained. The charge was then found to have formed a scaffold across the crucible. It was found impossible to smelt down the semi-fused mass forming the scaffold with the small electrodes at hand; the furnace was therefore allowed to cool and the material picked out.

The metal obtained in tapping had a coarse crystalline structure and was extremely hard and tough.

A polished section etched with picric acid and magnified 180 diameters showed irregular branching figures of a bright white constituent in a ground of granular pearlite.

Analysis:

C	0.71 percent
Si	2.14 "
(Large amount of scale in sample.)	
P	0.04 "
S	0.025 "
Mo	1.95 "

Charge: *Run No. 8.*

Iron ore	1,512 gm.
Coke	326 "
Lime	206 "
Calculated iron content	1,000 "
Calculated Mo. content	5 percent
Length of run	60 min.
Mean amperes	300
Mean volts	25
K. W.	7.5
Electrode consumption	65.0 gm.
Metal tapped	—

The furnace was started on a small amount of wrought iron punchings, covered by a little powdered coke. These melted rapidly, and a small charge was thrown in in small portions with the arcs running freely. When the metal was all melted, the entire charge was added and the electrodes embedded in the cold material. The small rods employed in experiment 7 had been replaced by full-size electrodes.

Reduction of the charge was rather slow. At the end of sixty minutes an attempt was made to tap. Slag flowed out freely and filled the mould, but the metal would not flow. The tap-hole was closed and a strong arc started to melt the mass of metal. Upon tapping, a portion of the metal was obtained. This was tough and malleable and had a very fine-grained structure.

The molybdenum mixture was added as usual just before reduction was complete. When the arc was started on the surface of the metal after tapping the slag, a white sublimate formed on the electrodes and the under surface of the cover. This was

probably MoO_3, formed by the oxidation of the metallic molybdenum in the steel, which was unprotected by slag.

A polished section of the metal tapped, when etched with picric acid and examined under a magnification of 180 diameters, showed a coarse network of the characteristic bright white constituent in a mass of pearlite.

Analysis:

C	0.92 percent
P	0.043 "
Si	1.10 "
Mo	2.15 "
S	0.036 "

Charge: *Run No. 9.*

Iron ore	756 gm.
Coke	163 "
Lime	103 "
Calculated iron content	500 "
Length of run	60 min.
Mean amperes	300
Mean volts	25
K. W.	7.5
Metal tapped	—
Calculated molybdenum content	0.6 percent

The furnace was started on the metal remaining in the crucible. When the crucible was well heated, the charge was added, and reduction was rapid. At the end of sixty minutes an attempt was made to tap. Slag flowed out freely, but the metal would not flow. This was due to a dam of metal formed just outside the tap-hole from a leak in the clay plug.

The furnace was allowed to cool, and a mass of metal weighing about 450 gm. was taken from the crucible with tongs. This metal was very malleable and tough and could be easily sawn and cut.

A polished section, etched with picric acid and examined under a magnification of 180 diameters, showed the characteristic network of double carbide and in addition a mass of needles of a white constituent resembling the carbide.

Analysis:

C	0.54 percent
Si	1.65 "
Mo	0.45 "
P	0.07 "
S	0.05 "

Run No. 10.

Molybdenum steel from molybdenite concentrates.

Analysis of concentrates (Woods Flotation Process):

Mo	11.10 percent
Cu	0.72 "
S	15.00 "
SiO_2	65.00 "

Molybdenum mixture (equivalent to 0.66 percent Mo in calculated iron).

Concentrates	100 gm.
Si-Fe	9.28 "
Lime	65.00 "

Charge:

As in previous runs, equivalent to 1,500 grams calculated iron.

Length of run	40 min.
Mean amperes	250
Mean volts	25
K. W.	6.25
Metal tapped	1,370 gm.

Furnace started on metal in bottom. Charge added in two portions and reduced rapidly. A tap was attempted after the addition of the first charge, but the metal would not flow. The molybdenum mixture was added in two equal portions and was reduced almost instantly. The tap was clean and the ingot obtained was sound and free from large blow holes. The increase in molybdenum content in the metal over that calculated was probably due to a concentration of the molybdenum in the metal reduced.

A polished section, taken from the center of the ingot, when etched with picric acid and examined under a magnification of 180 diameters, showed a fine network of the double carbide and small isolated patches of the same constituent all in a ground mass of pearlite.

Analysis:

C	0.70 percent
SiO_2	1.20 "
Mo	0.70 "
P	0.10 "
S	0.45 "

Conclusions.

Since the results of the experiments herein described, with the exception of runs Nos. 5 and 10, were qualitative rather than quantitative, further experiments would be necessary in order to determine the loss and distribution of molybdenum.

Since no analyses were made upon the slag, it is impossible to determine what proportions of the sulphur were removed by the ferro-silicon and the slag.

(1) Molybdenum steel can be made in the electric furnace by the direct reduction of iron ore and the addition of molybdenum in the form of molybdenite, MoS_2.

(2) Molybdenum steel of low sulphur content can be produced from molybdenite by the use of ferro-silicon as a desulphurizer.

(3) Molybdenum steel of low sulphur content can be produced from molybdenite in the form of low grade concentrates by the use of ferro-silicon as a desulphurizer.

With regard to the design of a furnace for small scale operations, the fore-going experiments would indicate that:

(1) The Girod principle as used was superior to the Heroult.

(2) A tilting furnace would be more effective than a stationary furnace.

(3) The tap hole on a stationary furnace should be made short, with a steep inclination. Ample provision for heating the tap hole should be made.

(4) Tar does not make a satisfactory binding material for crushed magnesite.

DISCUSSION.

Mr. F. A. J. FitzGerald: Mr. President, I read over this paper with a good deal of interest, giving as it does a very careful description of experiments, but I think it is rather objectionable to draw such basic conclusions from experiments with small furnaces that tap only about a thousand grams of metal.

There is another objection to the paper. There is a reference

to Girod principles and Heroult principles. One of the furnaces that these gentlemen used had two electrodes connected in parallel, the other terminal of the furnace being formed by the bottom of the hearth. Why should this be called a Girod furnace? Then, in another way of using the furnace they connected the two electrodes in series and the current passed through the charge. Why should this be called a Heroult furnace? This sort of thing encourages a rather objectionable electrometallurgical mythology.

Mr. C. A. Hansen: May I ask Mr. FitzGerald to suggest two names or adjectives that will make one's meaning as clear as the terms Heroult and Girod? They have a perfectly definite meaning nowadays and a single word conveys the equivalent of a paragraph of descriptive matter. The only objection, that I see, is that these terms, by inference, attribute invention of the two furnace types respectively to Heroult and Girod. If Mr. FitzGerald will substitute for these additions to our technical vocabulary on the order of "pinch" and "squirt" as applied to furnaces I don't doubt we would all accept them.

Mr. FitzGerald: Mr. Hansen is obviously a mythologist.

President Whitney: Mr. FitzGerald has raised a point that we ought to take care of. We must not forget that Siemens was the first to do these experiments in the general arc type of furnaces—I do not think I am saying anything that is not so—and if we speak of those furnaces by using the name of some man it seems to me it is well to discriminate with due regard to historical accuracy. I do not want to let this point go by without saying that I like to see these small experiments described. These little fellows who cannot tap the metal because they haven't enough to flow in one run show us the way we have to start almost everything. They realize that this is not a big, practical experiment, and it is something for them to come to us and present the report of their experiments. I think we do not want to do anything to discourage them, but rather everything to encourage them and help them along.

A paper presented at the Twentieth General Meeting of the American Electrochemical Society, in Toronto, Canada, September 21, 1911, President W. R. Whitney in the Chair.

RECENT PROGRESS IN ELECTRICAL IRON SMELTING IN SWEDEN.

By Thomas Duncan Robertson.

On May 6, 1909, a paper was read before this society by Dr. Eugene Haanel on the Electric Shaft Furnace of the Aktiebolaget Elektrometall of Ludvika, Sweden. He showed how three Swedish engineers, Assar Grönwall, Axel Lindblad and Otto Stalhane encouraged by the results of the Sault Ste. Marie experiments of 1907, devoted their energies to making the electric smelting of iron ores a commercial success. These three gentlemen formed a company called the Aktiebolaget Elektrometall to which the patent rights were assigned.

Assistance was given to them by the Swedish iron masters for the experimentation, which finally resulted in the construction of a 700 h. p. furnace at Domnarfvet in the autumn of 1908, a trial run which is fully described in Dr. Haanel's paper. The important point proved by this trial run was that regular smooth working was to be expected from this type of furnace.

This 700 h. p. furnace has since given great satisfaction and has produced many tons of excellent pig iron.

Realizing, however, the importance of thoroughly testing the furnace on a large scale, Jernkontoret, an association of Swedish ironmasters founded over one hundred and eighty years ago, determined to build at Trollhättan a furnace of 2,500 h. p. capacity.

Although having accumulated large funds they had not previously embarked in a commercial or technical undertaking, but of such moment to the Swedish iron industry was this question, that they voted a sum of $90,000 to put up the plant and to further develop the process.

The Swedish Government to assist in the undertaking, decided to furnish power at a nominal figure from their Trollhättan

station, an illustration of which is given in Fig. 1. There are at present only four of the projected eight sets running, each of which furnishes 12,500 h. p., three-phase current of 25 cycles at 10,000 volts tension.

One of the high tension transmission lines, and a railroad track, runs close to the site selected for the plant on the government property at Stallbacka, which is at the disposal of Jernkontoret until October 1, 1912. Two side tracks were laid, one

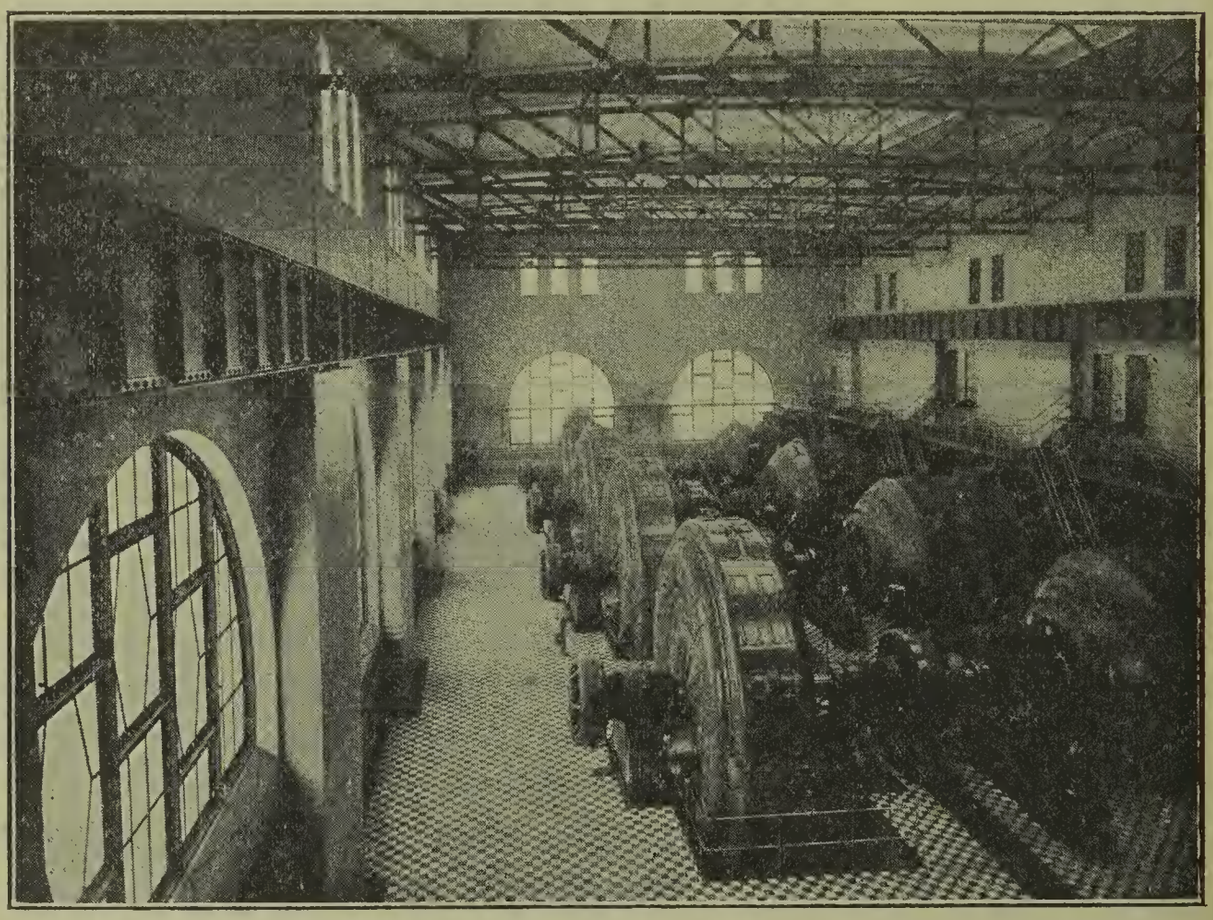


Fig. 1.

for the transport of fuel to the fuel storage house and the other for transporting other materials to, and iron and slag from, the furnace house. The general arrangement of the furnace house is shown in Fig. 2 and that of the fuel store in Fig. 3.

The fuel used is charcoal which is taken from the cars to the storage house by two band conveyors of cold rolled steel. Provision is made for the storage of about 7,000 cubic metres of charcoal, corresponding to a six months supply. From this store the charcoal is elevated to the furnace top (Fig. 4) by means of iron buckets running on a wire rope.

The ore is taken from the heap in barrows which are dumped outside the crusher building into an elevator truck which brings the ore up to an incline, along which it slides into the crusher which is driven by a 40 h. p. motor. The crushed material falls

Fig. 2.

Fig. 3.

into a sheet iron hopper, in which a sufficient quantity is collected to fill the truck to be elevated to the furnace top. The limestone, which is used as a flux, is crushed and treated in the same way as the ore.

The construction of the furnace is shown in Fig. 6, two separate portions are to be distinguished, viz.: the shaft and the crucible or hearth. The shaft is simply a shell of steel plate lined with firebrick. At the top it is riveted to an octagonal channel iron ring, which bears on two built up beams, these being in turn supported by the walls of the building. At the top of the shaft is a Tholander charging bell, a type of bell peculiar to Sweden which is specially designed to deliver the ore round

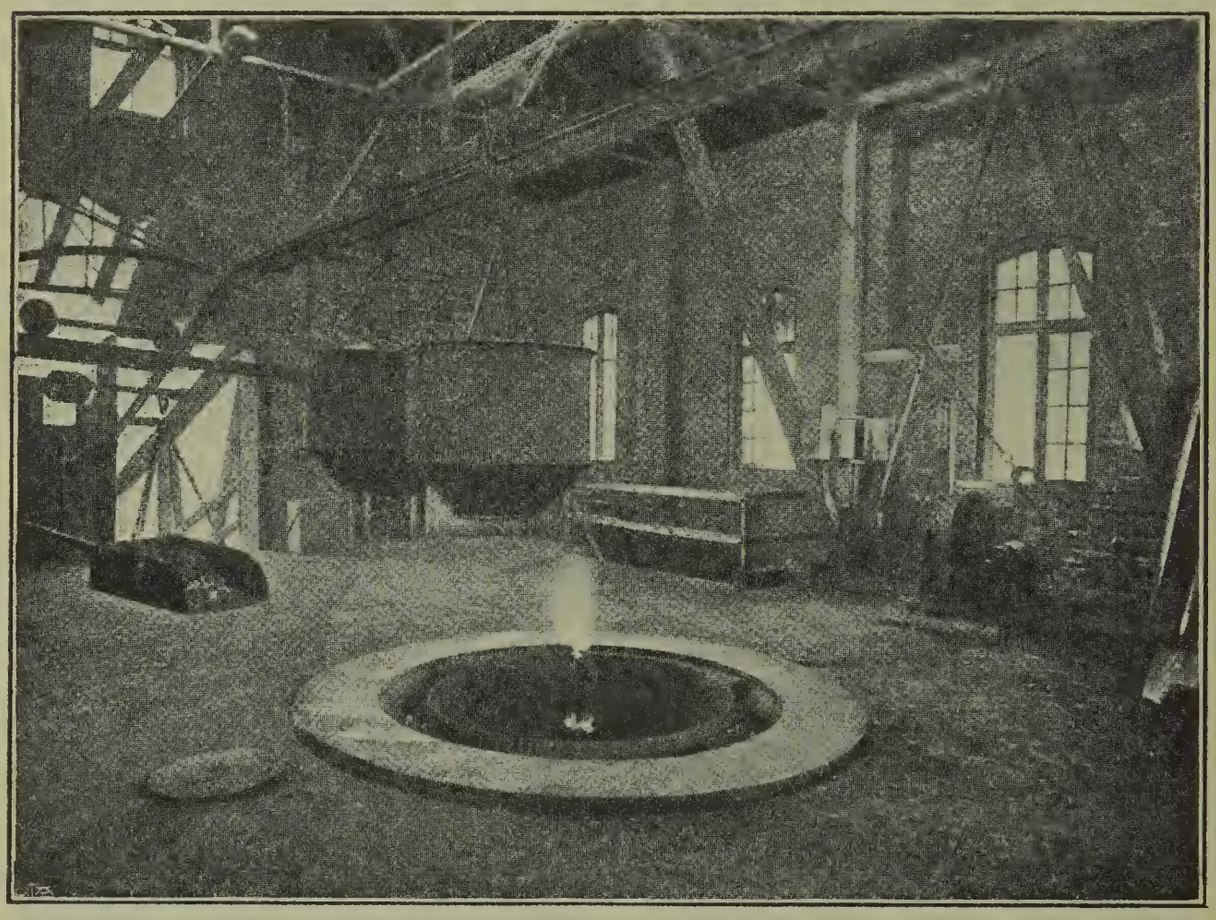


FIG. 4.

the sides of the shaft and the charcoal in the center. A 2½-h. p. reversible 3-phase motor raises and lowers the bell.

The crucible rests on a concrete foundation. It also has a steel shell, next to which is a lining of firebrick, with an inner lining of magnesite brick, while the bowl-shaped bottom is formed by ramming in a mixture of magnesite and tar.

The arched roof is built of firebrick and has four openings to admit the electrodes. These project through the roof at an angle of 65° to the horizontal. At the opening in the roof each

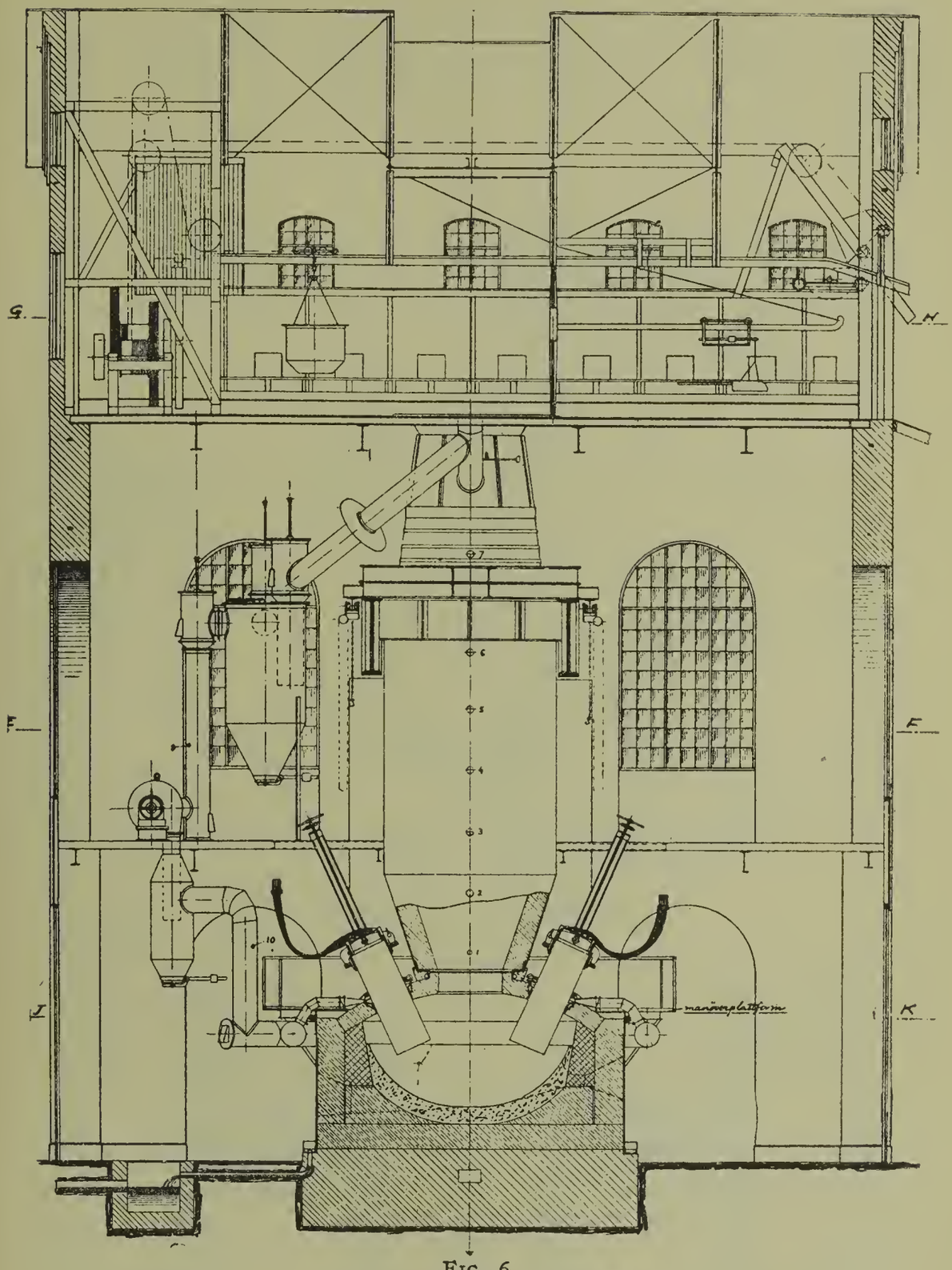


FIG. 6.

electrode is surrounded by a copper water jacket, provided with asbestos packing to prevent the leakage of gas. The electrical contact pieces are wedged between the upper end of the electrodes and a holder of cast steel which is supported in a frame, that can be raised or lowered between two guides, one on either side of the electrode.

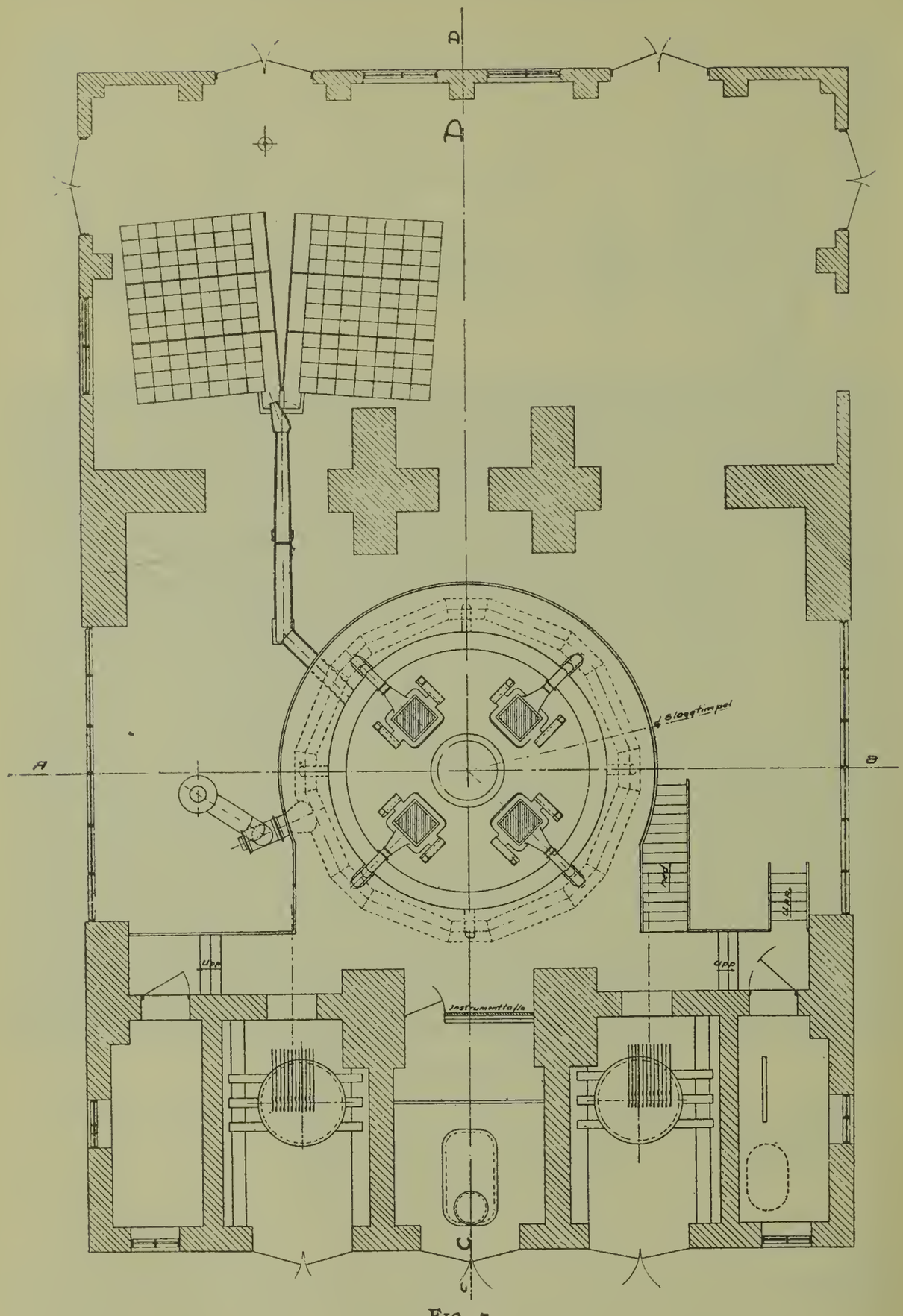


FIG. 7.

An important feature of the furnace is the provision made for the circulation of the gases produced by the reduction of the iron ore. The gas is drawn from the cool upper part of the furnace

by means of a fan, and blown under the hot roof of the crucible, serving the double purpose of prolonging the life of the roof, and at the same time giving up its heat to, and helping in the reduction of, the charge. The arrangement of the gas pipes is

A—Ammeters.
B—Electrodes.
C—Automatic Oil Switches.
D—Lightning Arrester
E—Induction Coil.
F—Kilowatt Meters.
G—Kilowatt Hour Meters.
H—Maximum Relays.
J—Switch.
K—Oil Resistance.
L—Main Switches.
M—Clock Control.
N—Fuses.
O—Transformers.
P—Transformers.
Q—Transformers.
V—Voltmeters.

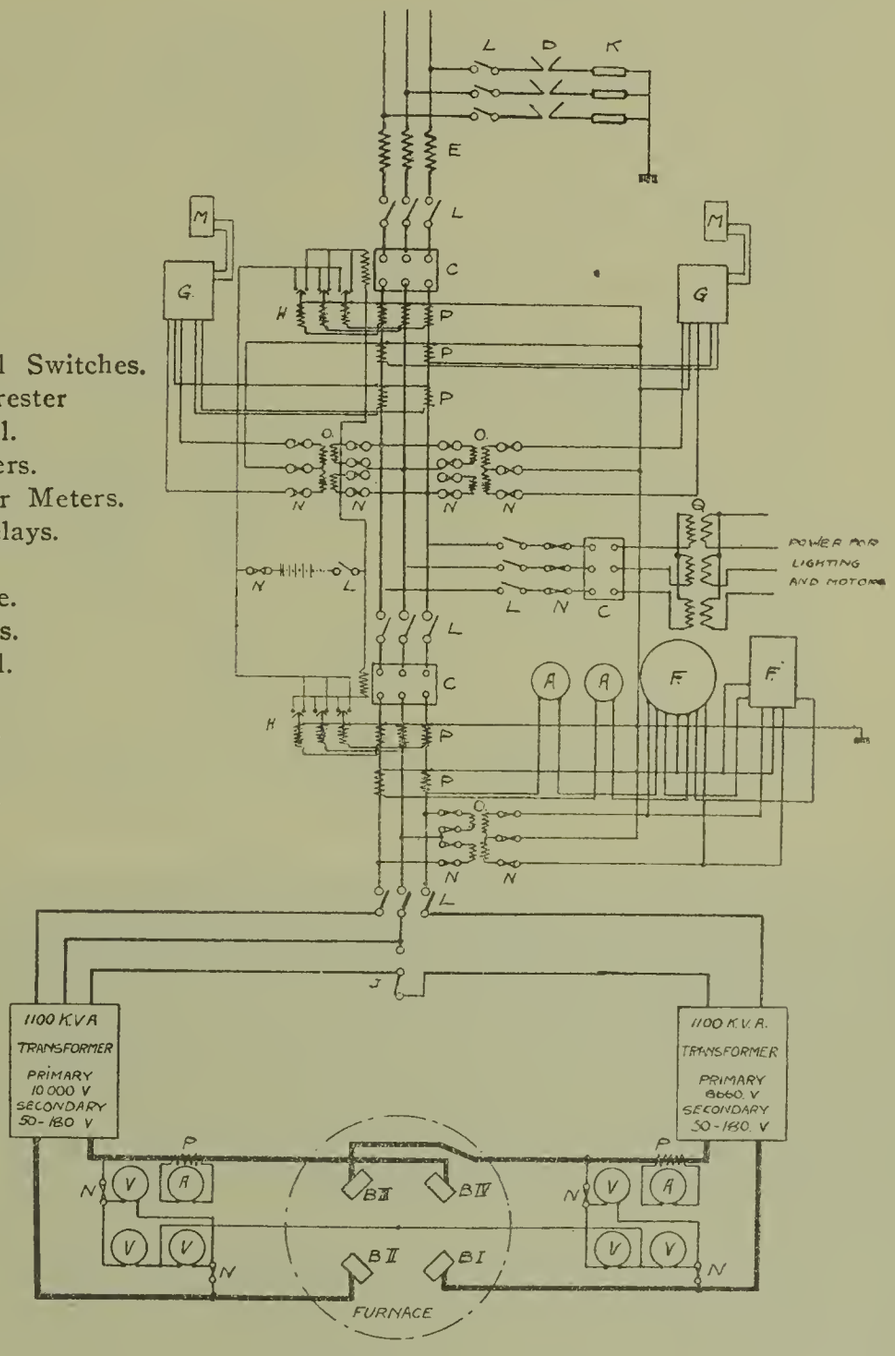


FIG. 9.

seen in Fig. 6; the four tuyeres through which the gas is blown being situated midway between the electrodes.

The tap holes, runners, and pig iron are those of ordinary Swedish blast furnace practice, the pigs are flat and rectangular and are cast in cast iron moulds, the general arrangement being

shown, in plan, in Fig. 7. During a large portion of the time the charge has been high in iron and the quantity of slag correspondingly small, so that the iron and slag have been tapped out together, and by means of a small dam the slag has been allowed to run off down a sand trench.

The transformers, electrodes, the tap hole arch and the walls of the hearth are water cooled. The water is pumped from the adjoining canal to a tank on the furnace top, about 100 gallons per minute being used.

The electrical arrangements are shown in Fig. 9. It will be seen that the high tension mains are provided with lightning arresters. The main switch has automatic circuit breakers operated by Siemens type relays adjustable for time as well as for overload. After the various series transformers are passed the current is taken through another automatic oil switch, to the furnace transformers. These are two in number each of 1,100 k. v. a. capacity, oil insulated, with water cooling. By means of Scott connections they transform the 3 phase, 10,000 volt supply to a 2 phase current the tension of which by altering the number of primary windings can be regulated between 50 and 90 volts. This alteration is carried out whilst the furnace is working by simply turning a handle, and the arrangements are such that the two phases can work at different tensions.

The power for lighting and for the various motors is furnished by small transformers giving 170 k. v. a. at 190 volts.

The instrument board has the following instruments: Two ammeters, two voltmeters, one direct "Ferraris" kilowatt meter, four voltmeters for the electrodes and two ammeters for the low tension bus-bars, the whole being fitted with the necessary cut-outs, etc. In a special instrument room on the next floor, there are two integrating kilowatt meters and one registering kilowatt meter.

The low tension bus-bars from the transformers to the furnace room are 24 in number, each 8 x 200 mm. cross section. From these bars to the electrodes the current is taken by flexible bare copper cables. (Figs. 8 and 10.)

An elaborate system of pyrometers is installed, the holes in the shaft for the thermo-elements, being marked in Fig. 6. Ordinary water manometers are permanently connected to the

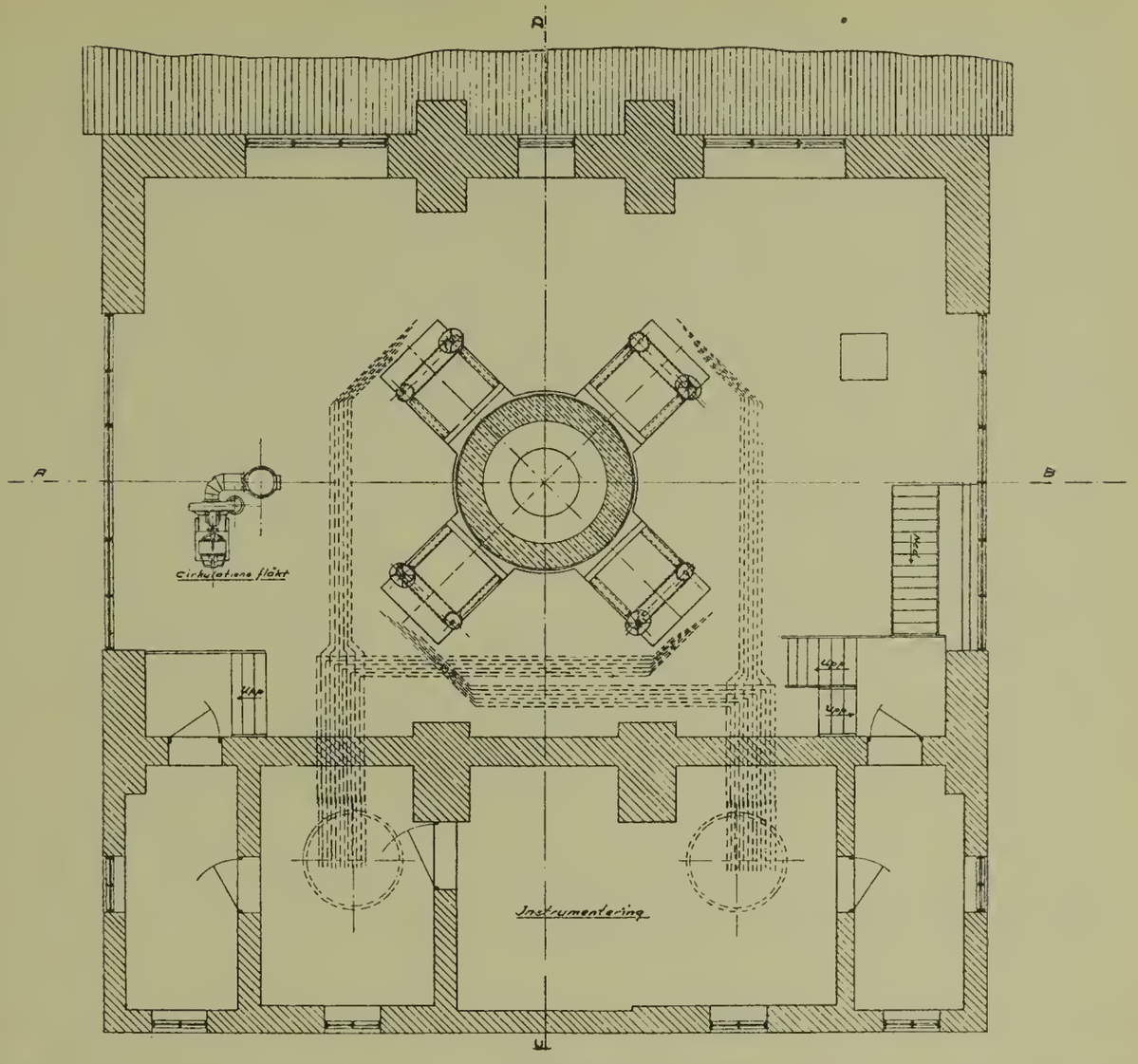


FIG. 8.

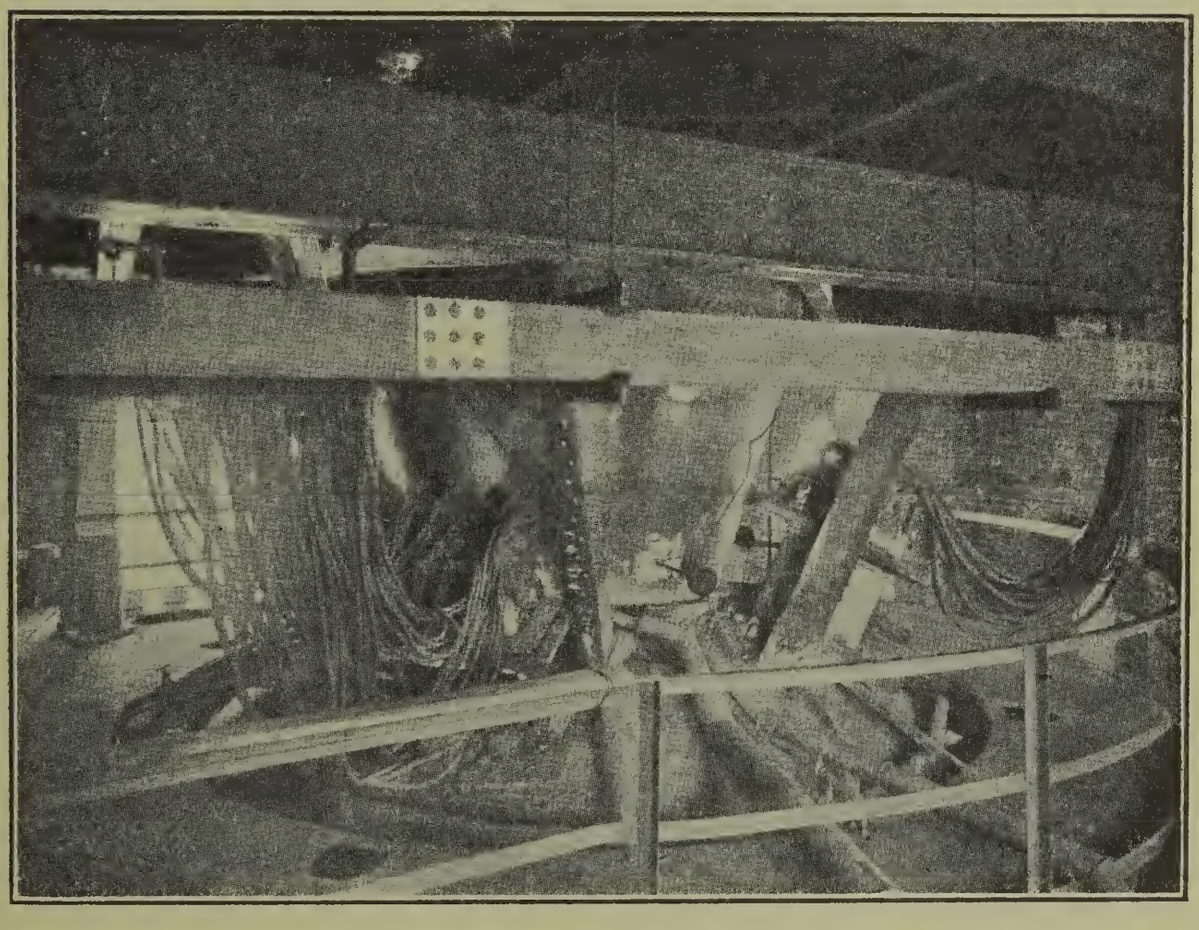


FIG. 10.

furnace top, the discharge side of the fan and to a hole in the roof of the hearth. A. "Mono" apparatus constantly records the percentage of CO_2 in the gas from the fan. The laboratory is fitted with gas analysis apparatus, and is well equipped for the analysis of the works materials and products.

The design of the furnace was based on the experience of the inventors at Domnarfvet. The calculations assumed a production of 7,500 tons of pig iron per year of eleven working months, or 23 tons per twenty-four hours. A cubic metre of charcoal was taken as weighing 150 kg., 3 kg. of pig iron being obtained from 1 kg. charcoal. In deciding on the correct volume for the furnace it was assumed that half the ore would be contained in the interstices of the charcoal and that the other half would require extra space, and also that the ratio of the volume of charge per day to the furnace volume is equal to 1.55.

It was then found that 23 tons of pig iron correspond to a volume of 51.1 cubic metres of charcoal and an extra space of 7.9 cubic metres for the ore, making a total of 59 cubic metres. Therefore the volume of the furnace should be

$$59/1.55 = 38 \text{ cubic metres.}$$

The diameter of the neck was the first dimension to be decided upon and this was fixed at 1,200 mm. It appeared desirable to make it larger but this would have necessitated a larger hearth to make room for the electrodes. The shape and depth of the hearth were based on the Domnarfvet experience. A high and narrow shaft would give a good gas reduction efficiency, but would mean a large back pressure against the gas, and a higher cost of construction. A medium was struck at 13.7 metres from the ground level to the furnace top.

At the time the furnace was designed, carbon electrodes in one piece of the necessary dimensions could not be obtained, so that electrodes were built up of 4 pieces, 2 metres long and 330 mm. square. Before being bound together the sides forming the joints were planed true by hand and the joint was made with a thick paste of graphite and molasses. The upper part of the electrode is covered with asbestos millboard and thin sheet iron, to prevent loss of heat.

The furnace was thoroughly dried out with wood and char-

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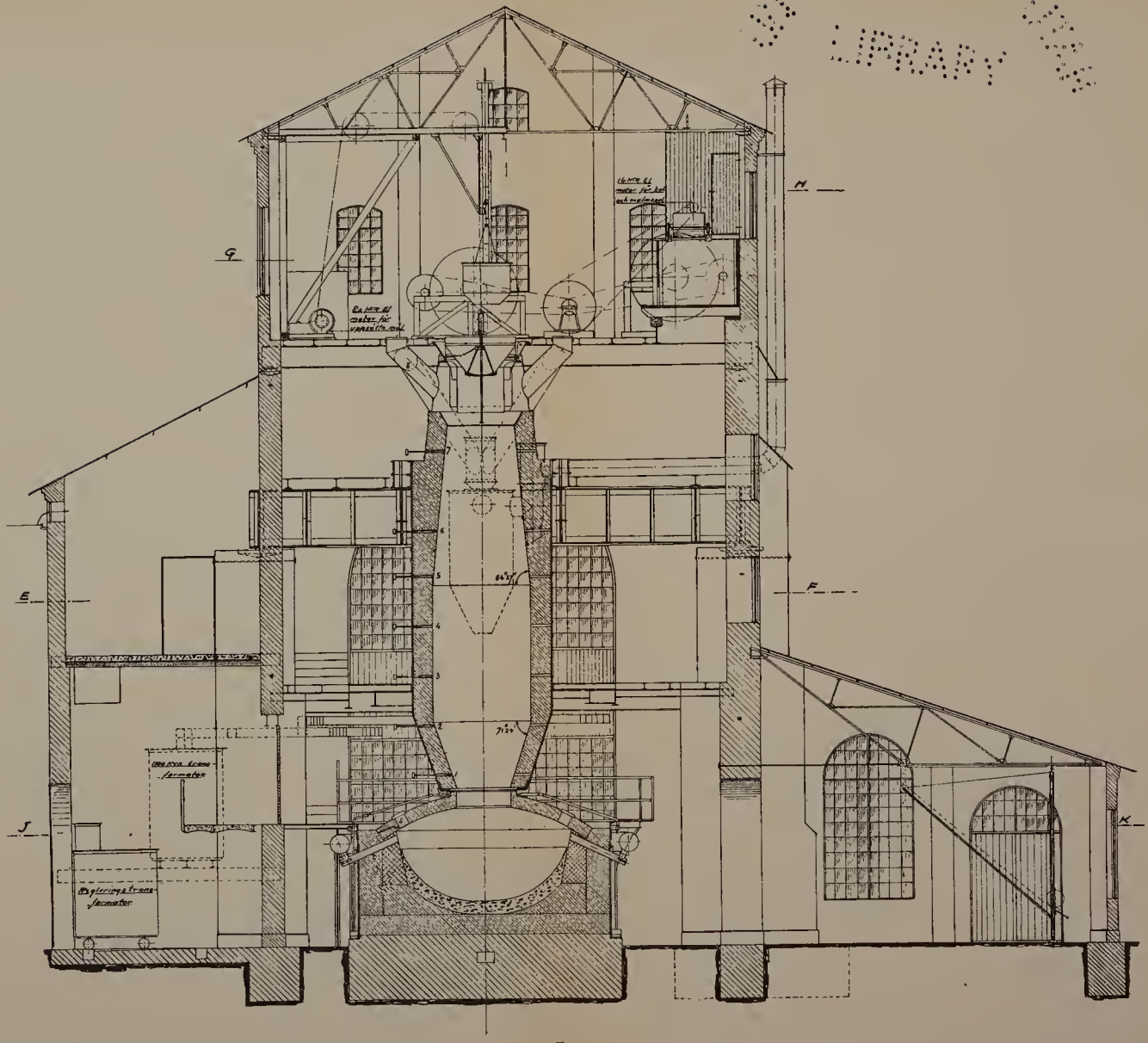


FIG 5.

coal fires, and heated up electrically by filling the hearth with coke and turning on the current.

Charging was commenced on the 15th of November, 1910. The furnace began to produce iron regularly and without difficulty, the first few tappings being rather high in sulphur from the large quantity of coke in the hearth.

The various iron works of Sweden sent their ores to be electrically smelted. In order to gain as much information as possible, the burden of the furnace was constantly altered to vary the grade of iron produced. Such treatment involving the frequent alternation of acid and basic slags was not good for the furnace hearth, and it must be admitted that it was something of an achievement for this to stand six months of this treatment without needing any serious repairs.

In Table I is given a summary of working results for the period November 15, 1910, to April 9, 1911.

TABLE I.

	Period of Operation					
	1910 Nov. 15 *	1910 Nov. 16 to 1911 Feb. 11	1911 Feb. 11 to Feb. 19	1911 Feb. 19 to Mar. 19	1911 Mar. 19 to Apr. 9	Total
Percent of Iron in Ore	64.92	65.57	65.06	49.50	57.92	61.54
Percent of Iron in Charge	59.80	62.10	62.56	42.42	53.06	57.00
Slag per ton of Iron (kg.)	390.	205.	224.	780.	458.	327.
Material charged per hectolitre of Charcoal (kg.)		66.49	71.13	90.31	69.88	70.77
Charcoal per ton of Iron (hectolitres)		24.22	22.47	26.10	26.97	24.79
Charcoal Contents:						
Water (kg.)		69.1	50.8	59.8	40.2	60.9
Gas "		41.7	36.9	49.3	43.1	42.9
Ash "		11.8	11.0	13.2	17.2	12.8
Coke "		293.1	277.6	323.4	325.7	301.4
Total kilogrammes		415.7	376.3	445.7	426.2	418.0
ELECTRIC POWER	h. m.	h. m.	h. m.	h. m.	h. m.	h. m.
Time consumed working	7 50	2,009.56	184.32	639.18	506.34	3,348.10
Time consumed in interruptions		105.39	4.58	20.57	22.11	153.45
Total time	7.50	2,115.35	189.30	660.15	528.45	3,501.55
Average load (kw.)	1,121	1,319	1,694	1,017	1,733	1,344
Total kw. hours used	8,780	2,651.029	312,601	650,480	877,706	4,500,596
Kw. hours per ton of Iron	3,800	2,296	2,149	2,623	2,643	2,391
Iron per kw year (tons)	2.31	3.82	4.08	3.34	3.31	3.66
Electrode consumption:						
Gross (kg.)		13,012	1,578	2,281	2,474	19,345
†Net "		6,743	763	1,121	1,285	9,912
Per ton of Iron:						
Gross (kg.)		11.24	10.84	9.19	7.45	10.28
Net "		5.83	5.24	4 52	3.87	5.27

* Filling up of furnace. †Net = 51.24 percent of gross.

As might be expected the output per kw.-year is proportional to the percentage of iron in the ore, but with ores producing a greater quantity of slag the electrode consumption is smaller.

The thermal efficiency of the furnace was carefully worked out, when it had been running for five months, after which time the lining had worn rather thin, so that radiation losses were probably higher than normal. However, 80 percent (the figure obtained) compares favorably with 82 percent for the efficiency of the Swedish charcoal blast furnace, and with the 70 percent of the English blast furnace.

As the volume of the furnace and gas piping is constant an excess of gas is being continually produced, proportional to the amount of ore reduced. This gas is allowed to burn into the air at present at Trollhättan, but in the newer plants arrangements are to be made for using it for calcining purposes. About 550 cubic metres of gas are produced per ton of pig iron, and each cubic metre will develop 2,100 to 2,500 Calories, as the gas averages about 80 per cent. of combustibles.

When calculating the various charges the quantity of charcoal is kept constant at 6½ hectolitres and the amounts of ore and limestone are varied to produce the desired grade of iron, a typcial charge at the time when the author was present at the furnace being:

Magnetite 266 kg.		
Concentrates 133 kg.	410 kg.	
Mill Cinder 11 kg.		
Limestone	32 kg.	
Charcoal 6½ hectolitres	or 100 kg.	

This is dropped into the furnace at one raising of the bell and tappings are made every 18 charges, or about once every six hours.

The conditions governing the grade of iron produced are similar to those in the ordinary blast furnace, except that the irregular influence of the air blast is absent. The furnace gives the maximum output when making white iron, as the making of grey iron requires a rather higher temperature and consequently a greater power consumption. By increasing the amount of ore in the

charge when the furnace is making white iron, a low carbon iron with very little silicon is produced, a typical analysis of which is as follows:

	C.	Si.	Mn.	S.	P.
Percentages	2.60	0.10	0.11	0.02	0.01

This iron naturally is full of holes, but instead of these having colored oxidized surfaces, they are silvery white; the absence of oxygen from the furnace atmosphere accounting for the production of this grade of iron free from oxides. The results of making steel from this iron are given later.

The tension employed varies greatly and depends mainly on the resistance of the charge. This broadly speaking increases with the amount of slag produced and with the wear of the electrodes but decreases as the amount of charcoal in the charge becomes greater. The maximum power allowed is 1,900 kw. and to avoid overheating the electrodes the current must not exceed 17,000 amperes, so that the voltage has to be regulated accordingly.

The wearing of the electrodes has given rise to some curious and interesting results. In Fig. 11 is shown a new electrode with its iron sheath and contact frame. When this is placed in the furnace, the bottom end projects into the actual charge and is gradually abraided and consumed. Between the stock line and the roof is a space filled with furnace gases which contain about 15 percent CO_2, so that the portion of the electrode exposed to them is very gradually oxidized. The result of this is shown in Fig. 12 where the half-consumed electrode was removed for inspection, the line of the surface of the charge being clearly visible. Fig. 13 shows the stump end of an electrode which had been lowered as far as possible into the furnace and kept there until unfit for further use. It will be seen that two of the four carbons which compose it have been burnt through to the center. About three weeks is taken to burn through an electrode above the stock line, so that in practice each electrode is lowered once every two weeks, to ensure regular working without risk of fracture, which would result in a lump of electrode being left in the charge. With the object of cooling the roof immediately behind the electrodes, gas was blown in beneath the water jackets during a portion of the time. The effect is shown in Fig. 14, the CO_2

in the stream of gas having burnt a hole in the electrode, causing this practice to be discontinued.

A point of interest from a Canadian point of view is the success met with in smelting magnetic concentrates at Trollhättan. The design of shaft in this particular case is not considered suitable

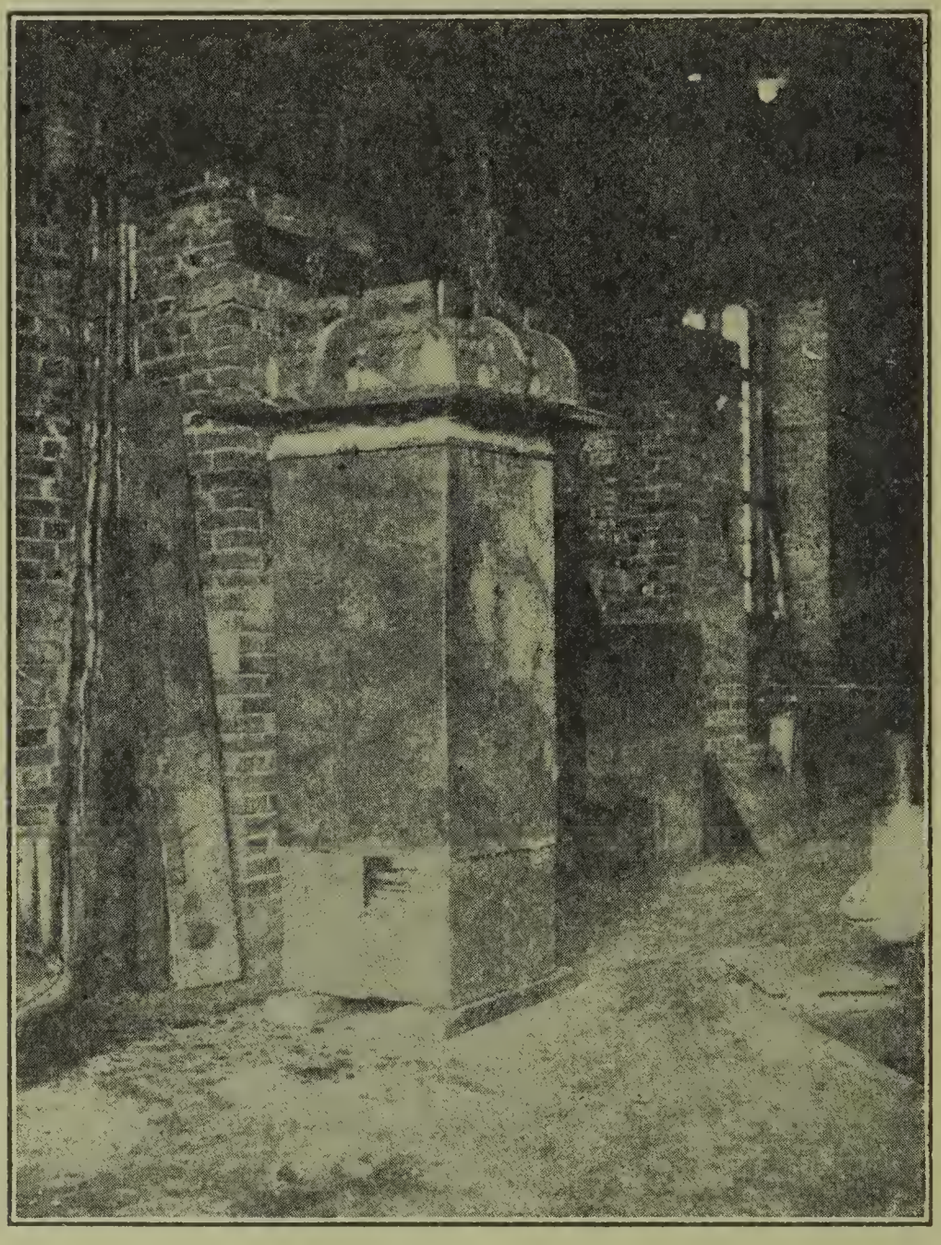


Fig. 11.

for the purpose, being too narrow, but in spite of this, 65 percent of finely-divided concentrates caused no inconvenience in working. The inventors of the furnace are of the opinion that, with a specially designed shaft, charges of all fine concentrates could be smelted. Canada is rich in deposits of iron sands and lean magnetites, which are easily concentrated, but which are expensive

to nodulize or briquette into a form suitable for blast furnace smelting, so that where these are within easy reach of water powers, there seems to be a good field open for electric smelting.

Dr. Haanel, in his report on the experiments made at Sault Ste. Marie, mentions that no difficulty was experienced in smelting

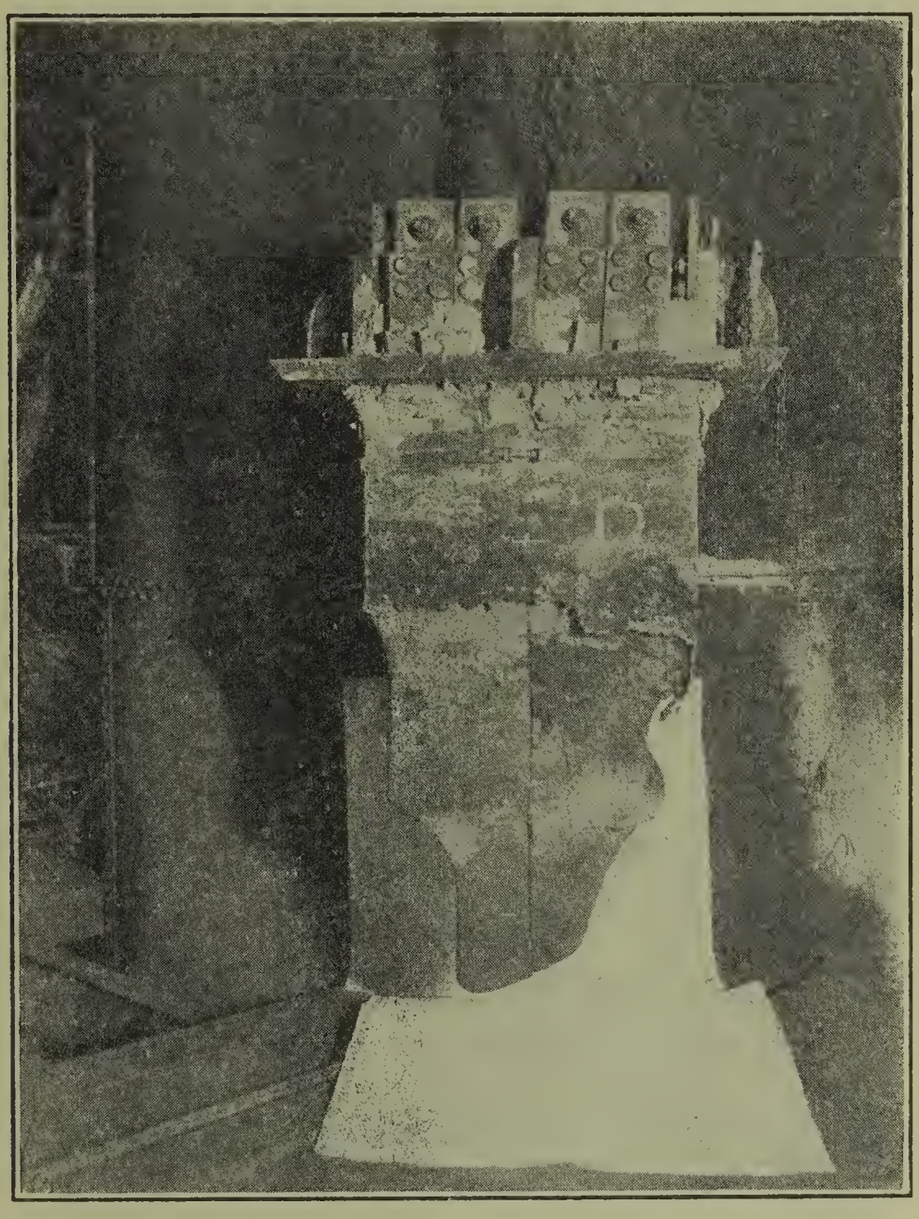


FIG. 12.

titaniferous ores electrically, and it is interesting that the Swedish experience bears out this point, although no ores were used with more than 0.8 percent TiO_2.

The electric smelting of ores with high sulphur content is another interesting problem. Dr. Haanel was successful in producing low sulphur iron from sulphurous ores, but in Sweden

there are practically none of these ores mined, so that this point was not confirmed on a large scale at Trollhättan; however, there can be little doubt that the electric furnace, with its reducing atmosphere and basic lining of the hearth, permitting as it does of the use of a slag very rich in lime, offers the best method of producing sulphur-free pig-iron from ores containing that unwelcome element.

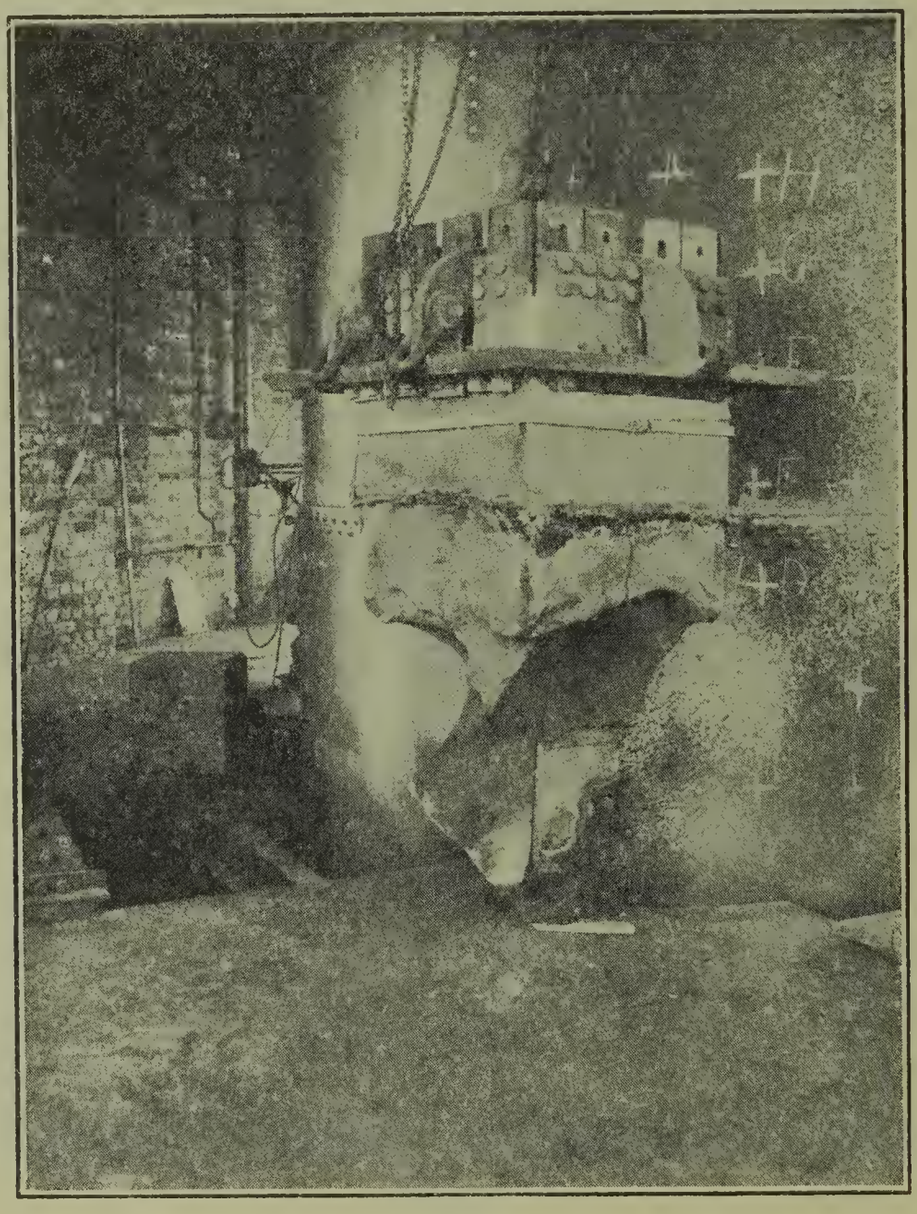


Fig. 13.

The pig iron produced by the furnace was sent to various Swedish iron works for conversion into steel in open-hearth furnaces. The characteristic feature of electric pig iron is its freedom from oxides; and in consequence electric pig of normal silicon content (say, 1 percent and over) takes a longer time and more ore to convert into steel than ordinary blast furnace grey

iron. Low-carbon electric pig iron, however, is found to give surprising results, charges made up of 50 percent of this iron and 50 percent of scrap producing hot fluid steel with considerable saving of time over ordinary practice. As was to be expected, the open-hearth furnace managers looked somewhat ask-

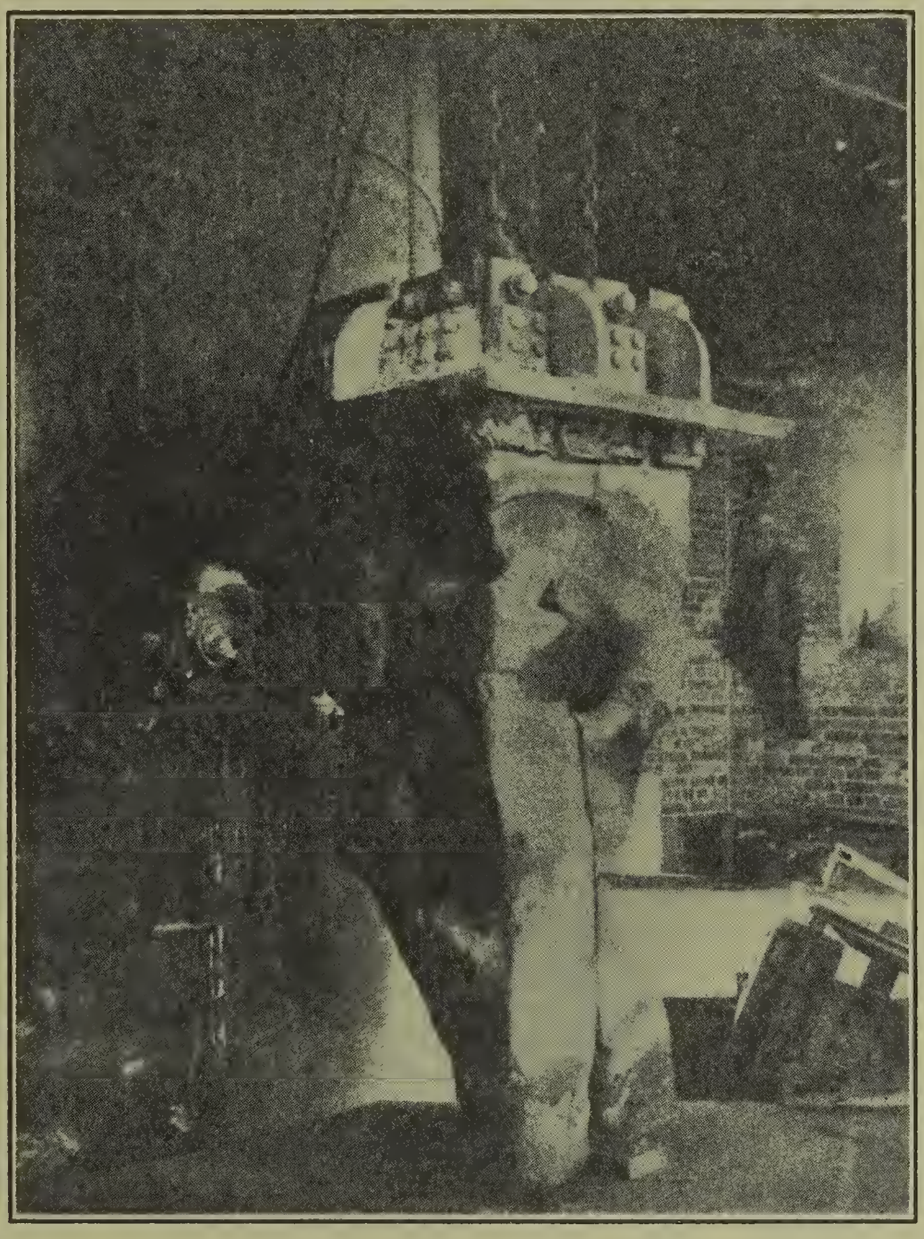


FIG. 14.

ance at this iron at first, as they knew the disastrous effect of using low-carbon iron full of holes from the blast furnace; however, after giving it a trial, the workmen asked for more, as they said that the furnace worked better and more rapidly with the new pig iron.

Fortunately for the electric furnace in Sweden, it is more economical to make the white iron, that the steelmakers prefer, than

to make high-silicon grey iron. Thus it may be maintained, on the strength of the experience gained, that for the open-hearth process, high-silicon contents are detrimental rather than advantageous, while in the blast furnace pig iron a certain quantity

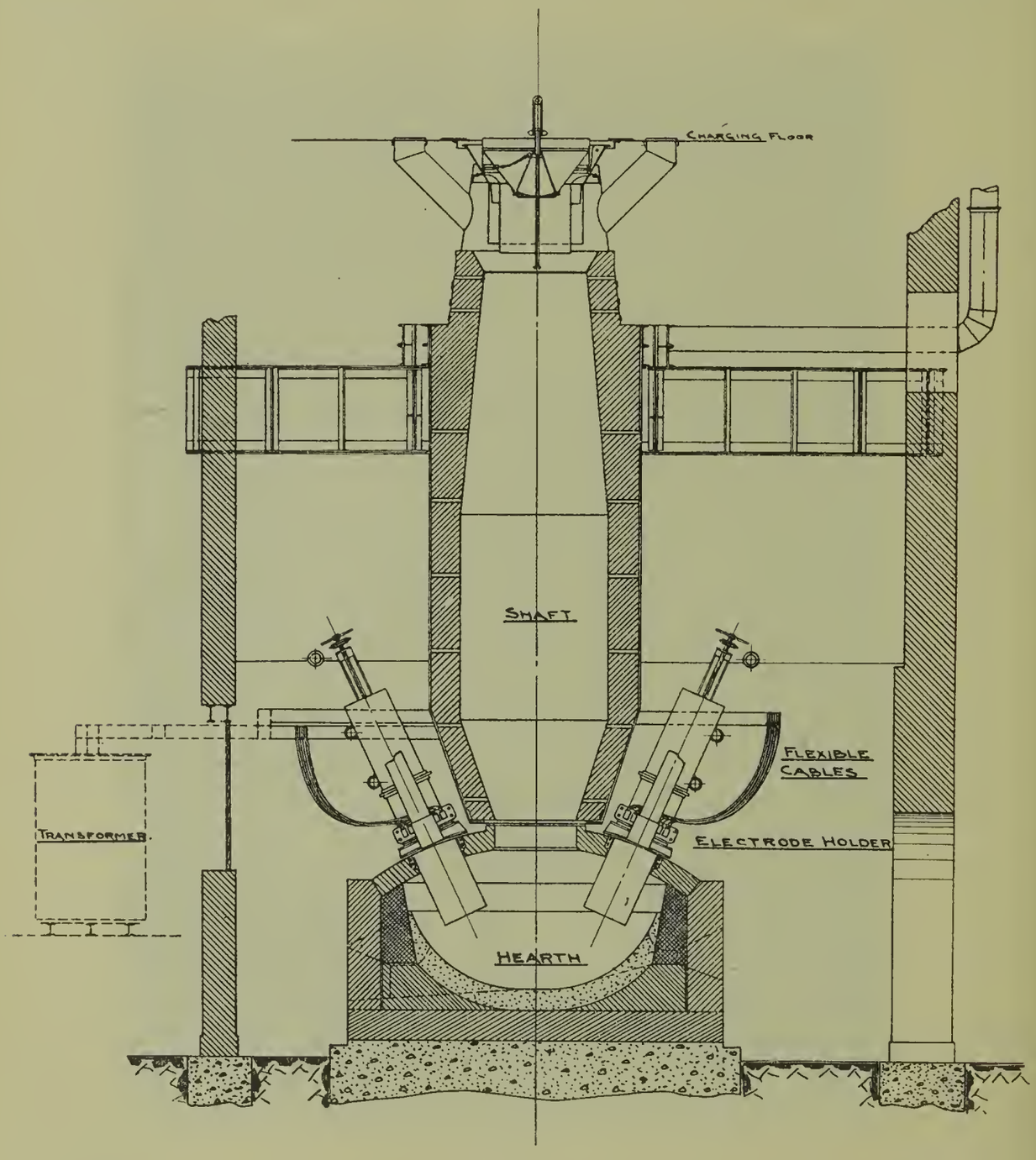


FIG. 15.

is necessary to neutralize the defects of a reduction process less perfect and ideal than that employed in the electric furnace.

The quality of the steel produced has been thoroughly tested by making it up into various products and comparing it with steel made from Swedish charcoal blast furnace iron. In no case was

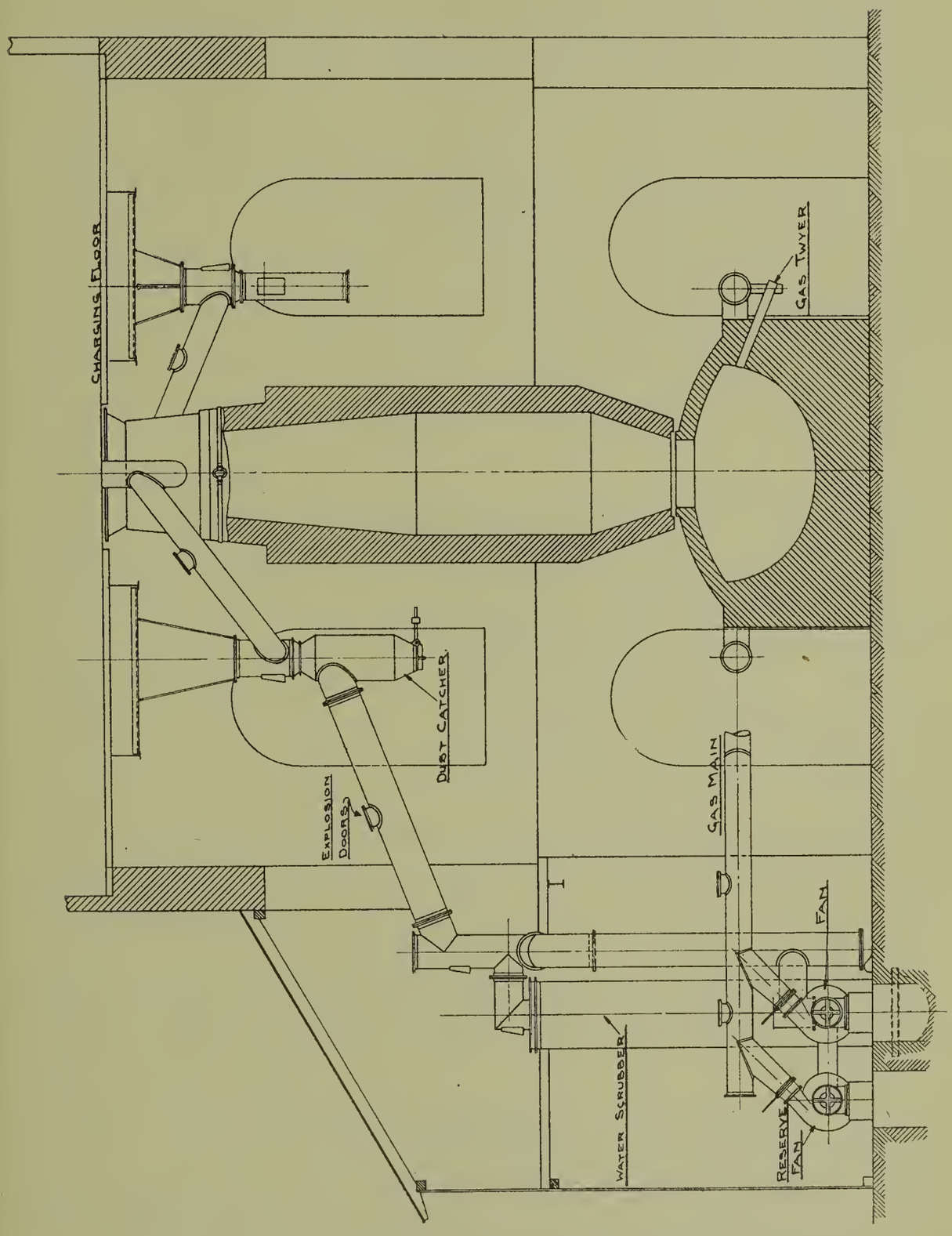

CHARGING FLOOR
GAS TWYER
DUST CATCHER
EXPLOSION DOORS
GAS MAIN
FAN
WATER SCRUBBER
RESERVE FAN

FIG. 16.

the new steel inferior, and, from the most recent reports to hand, it was in several cases decidedly superior.

The Trollhättan furnace was shut down at the end of May of this year in order to make certain alterations suggested by the results of its six months' campaign. Two of these deserve mention.

1. Since the furnace was designed, the manufacture of large carbon electrodes, of high conductivity, has made great progress. It is now possible to obtain cylindrical electrodes of 600 mm. diameter, fitted with screw joints. These have recently been installed at Trollhättan, and result in two considerable improvements, the first one being that the loss due to stump ends is done away with, as when an electrode becomes too short, a new one can be screwed on above it. The second one is that the electrical contact can be made at the point where the electrode enters the roof, in this way saving about 40 kw., which with the old arrangement was lost, due to the resistance of the whole length of the electrode through which the current had to pass. This new arrangement will permit of the furnace taking 3,000 h. p. instead of 2,500 h. p., and is shown in Fig. 15.

2. The other important alteration is in the gas-circulating arrangement, the new form of which is seen in Fig. 16. The old dust-catcher was not very effective, as only very fine dust is expelled from the furnace. The result was that the dust collected in the centrifugal fan, and had it not been that this was kept fairly clear by a jet of water, would have choked up the blades in a few hours; as it was the fan had to be stopped and cleaned every week. Now a water-scrubber takes the place of the dust-catcher, and a fan on the ground floor blows the cleaned gas into the furnace, and the other fan shown being held in reserve.

At the present time the following "Electrometall" furnaces are either working or in course of erection:

Sweden—Trollhättan, one furnace	3,000	H. P.
Domnarfvet, one furnace	4,000	"
Hagfors, two furnaces (3,000 H. P. each)	6,000	"
Norway—Tyssedahl (Hardanger), two furnaces (3,500 H. P. each)	7,000	"
Arendal, two furnaces (2,500 H. P. each)	5,000	"
	25,000	"

In these newer furnaces, 3-phase current is used, and by means of three single-phase transformers, low tension current is furnished to six carbon electrodes. Owing to the recent improvements in electrode manufacture and furnace design, it is now possible to construct units up to 6,000 h. p. which it is expected will work for two years without serious repairs to the hearth.

Rather more than three metric tons of pig iron are produced by the electric furnace per electrical h. p. year, using one ton of charcoal for the reduction of the ore. The blast furnace uses about three times this amount of fuel per ton of iron, so that in Sweden, where, owing to the demand of wood for the pulp industry, the price of charcoal is yearly advancing, there is a great field open for electrical smelting on account of the saving in fuel.

The furnaces in Norway have a somewhat different form of shaft, as coke is the fuel employed for smelting. The volume of the shaft is smaller, but its diameter is greater, than the corresponding shaft of a charcoal furnace. The coke in the charge gives a greater conductivity, so that a lower tension has to be used.

Summary.

1. Electric smelting of iron ores is no longer in the experimental stage, but in Sweden is thoroughly established on a commercial scale.

2. The smooth and regular working of the furnace at Trollhättan has been a marked feature, probably less trouble being experienced than would have been the case with a blast furnace doing similar work.

3. The quality of iron produced for steel-making purposes has been shown to be equal and in some cases superior to that of Swedish blast furnace pig iron.

4. The comparative costs of electric and blast furnace smelting depend in general on the cost of suitable fuel and electrical energy, as the electric furnace simply substitutes one electrical h. p. year for two metric tons of blast furnace fuel. In Sweden

there is no doubt that the electric furnace has come to stay on account of cheaper production.

5. The electric furnace is specially adapted to the smelting of finely-divided ores or concentrates.

6. Electric smelting in Sweden has a national value, as its adoption will result in the development of water powers which could not in many cases be profitably utilized for any other purpose.

In conclusion the author wishes to acknowledge his indebtedness to Messrs. Grönwall, Lindblad and Stalhane and their staff, and also to Messrs. Leffler and Nyström, of Jernkontoret, for drawings and information given to him in Sweden, without which this paper would never have been written.

Canadian Bövingh Co., Ltd.,
Toronto, Canada,
August 15, 1911.

DISCUSSION.

MR. WILLIAM BRADY: On page 377, Mr. Robertson refers to the crushed material. What size is it crushed to?

MR. T. D. ROBERTSON: The ore as mined in Sweden is received in cars, and there are pieces probably over one foot long. It is usually crushed to about the size of one's fist. The real reason that they have to crush it in that way is owing to their peculiar type of charging bell, which consists of two rings, the ore being put in the outer ring. The space through which it has to pass is only six or seven inches wide.

MR. BRADY: Then the reason for crushing is not because a larger piece would not smelt?

MR. ROBERTSON: No.

PRESIDENT WHITNEY: We owe a debt of gratitude to Mr. Robertson for presenting this subject. The matter of manufacturing pig iron by electric furnace methods is one of immense magnitude. We have been groping about in the dark, watching

the few who have been doing the work in this direction, but this is something which is likely to advance rapidly. A few years ago, I remember well, we thought it was foolish to talk about making pig iron by electric processes because of the immense cost of the electrical energy. It took us some time to learn that we could make tool steel by that process and make any money out of it, and now they are actually operating furnaces that are making pig iron which is the cheapest metal we have.

Mr. Louis Simpson: As there seems to be so much trouble with the dust and the gas, is it necessary to use the gas in the furnace? I understand that at Heroult, California, where they have a similar furnace, they are using no gas. I am also told that they operate their furnace in rather a different way so far as the electrodes are concerned; that whereas in the Swedish furnace the electrodes were placed for free burning, at Heroult the electrodes enter into the charge further, and, therefore they do not require any cooling effect from the gases. Now, it seems in the work done in Sweden, the gas from the furnace, where it enters the shaft, never or seldom exceeds 500° C., whereas in the blast furnace the heat, where it enters the shaft, is over 1,000° C. I think you will find that there is little or no reducing effect in the shaft of the furnace at the temperature obtained. In some experiments made in Sweden the average at the entrance to the shaft ran only from 682 to 241° C. It would seem to me that great loss is bound to ensue from the washing of those gases, and that when they go into the furnace again there is not only the loss from heat taken up by the water but also a loss arising from the water that the gas absorbs, followed by chemical troubles which will be readily understood by those making iron and steel.

I have heard from England that the buyers of the Swedish electric pig have found great trouble from the irregularity in quality. Has that difficulty been overcome? I received a letter from one of the largest dealers in Sheffield, in which he stated that he absolutely declined to handle any more electric pig entirely on that account. This is a very serious matter, because the pig iron made by the electric furnace is supposed to be of a higher grade than that made by the blast furnace, but if it is going to be irregular in quality, it will not bring the highest price.

MR. ROBERTSON: In answer to Mr. Simpson's questions, first of all, regarding the question of the gas used in the furnace, there has been a good deal of trouble with the dust, not only with the earlier arrangement of an ordinary dust-catcher, but even with this new arrangement for washing the gas. However, they are still making improvements, and they hope to overcome this difficulty before very long. The inventors of the furnace, I may say, attach great importance to this blowing of the gas in under the roof of the furnace, because they maintain that the important point is to make this furnace last as long as possible, and in order to do this they consider it absolutely necessary to have the roof cool.

Mr. Simpson mentioned the placing of the electrodes in the California furnace somewhat lower than in ours. Unfortunately, I have not the details regarding this furnace and I cannot say exactly what the effect of that would be; but regarding the reducing effect of these gases, I think the temperature about half way up the shaft is probably about 500° C., but the reduction of magnetite by carbon monoxide gas—and this furnace gas contains about 80 percent of that gas—takes place at as low a temperature as 300° C., so that in the shaft of this electric furnace there is considerable reduction of the ore although the temperature is not nearly so high as in the upper part of the blast furnace shaft.

As to the loss due to washing the gas, I have no information to give on that particular point, but no doubt they will have trouble from the very fact that their gas is wet as it must be after passing through the gas scrubber. However, they are doing a good deal of work on that point and hope before very long to have the gas circulation quite perfected.

Regarding the irregularity of the electric pig iron, I am afraid I cannot say very much. So far as I know, most of the pig iron made at Trollhättan was used up by the various iron-masters who were in the association which built the furnace, and of that pig iron, I do not think very much was offered for sale in England. The electric pig iron made previous to that could only have been made in the small furnace at Domnarfvet, and I do not know whether that was offered for sale in England or not. However, the pig iron offered for sale in England was probably

made in quite a small furnace, and since that time we have used larger furnaces and a more perfect method of working, so that I think we need have very little fear as to the regular quality of the Swedish electric pig iron. In Sweden, we must compare the electric pig iron with the Swedish blast furnace pig iron, and the steel makers of Sheffield experience a good deal of trouble from the irregularity in composition of this blast furnace pig iron. It is said that our electric furnace pig iron appears more regular in composition than the Swedish blast furnace pig iron.

Mr. Simpson: I am very glad to hear that statement made. I am a believer in the electric furnace and I could not see any reason why there should be such a great variation in quality. In the report of the experiments made at Trollhättan it appears that the highest temperature ever observed at the bottom of the shaft has been 682° C., and the lowest has been 241°; and that the temperature at the top of the shaft went from 205 to 29, with an average of only 76. I think that if the experiments made at Trollhättan have been properly reported, and if the report has been properly translated, we cannot expect that there will be much reduction in the shaft of the furnace.

Mr. Robertson: With reference to Mr. Simpson's remarks concerning the temperature of various points of the shaft I have referred to the Official Report of the Jernkontoret on the working of the Trollhättan furnace and find that the average temperature during the month of January, 1911, at the foot of the shaft (Point No. 1 in Fig. 6 of my paper) was 534° C., and that at Point No. 4 was 351° C. Unfortunately the readings at Point No. 5, rather more than half way up the shaft, were discontinued, but from the other figures given it would appear that the temperature at this point is not below 300° C., so that reduction of the charge by the gas, rich in CO, probably takes place throughout the whole of the lower half of the shaft.

Mr. M. M. Kohn: I should like to ask Mr. Robertson about the blowing of that gas in the furnace. Is it only for the purpose of cooling off the furnace, or is it also done to bring about the reduction of the ore?

Mr. Robertson: The object of the gas circulation is to cool the roof and at the same time aid in the reduction of the charge.

MR. F. J. HAMBLY: I would like to ask Mr. Robertson whether any difficulty is experienced in obtaining a pig iron containing from 3 to 4 percent of total carbon, similar to the ordinary blast furnace product. The conditions in the electric shaft furnace are very different from those in the blast furnace where a large excess of carbon is present, and I notice that in Mr. Robertson's paper low carbon pig iron containing from 2 to 3 percent total carbon is twice mentioned.

MR. ROBERTSON: At Trollhättan all grades of pig iron were made. I think they made iron with even less than 2 percent carbon; but on the other hand they made a great quantity between 2½ and 4 percent. This question of making a low carbon iron in the Swedish electric furnace has been very interesting, because they thought there was a great field open for the use of their low carbon iron in open hearth practice, but further experiments are to be made to test that point. There is no more difficulty in making iron with 4 percent carbon in the electric furnace than in the ordinary blast furnace. It is merely a question of how much charcoal you mix with your ore; but, on the other hand, the electrical power consumed as I pointed out in my paper, is less in the making of ordinary, normal white iron than in the making of gray iron, because the conditions are similar to those of the blast furnace, and a higher temperature and more charcoal is needed to produce gray iron than white iron. The analysis I gave is not to be regarded as a typical analysis of electric furnace pig iron; it is a typical analysis of the specially low carbon iron with which the interesting results in the open hearth furnace were obtained.

MR. CARL HERING: The author states that the thermal efficiency is 80 percent. How was that determined and which of the many efficiencies does he refer to?

MR. ROBERTSON: In answer to Mr. Hering, I am unfortunately unable to give you the exact details. This was calculated by the engineers in charge of the Trollhättan furnace but I do not think it was published in the report. They calculated it out just to get the information for their own use. They told me the method employed when I was over there, but unfortunately I did not make a note of it. I think it was worked out on the

same basis as they work out the efficiency for a charcoal furnace. I am sorry I cannot give you the details of it.

MR. F. A. LIDBURY: I would like to point out to Mr. Hering that on page 385, in Table 1, full details are given of the power consumed, slag per ton of iron and the percentage of iron in the charge, from which any one can calculate out whatever particular type of efficiency his own idiosyncrasies indicate.

MR. HERING: There is nothing said in this table about the power factor without which no true efficiency can be calculated. Have you any idea what that was?

MR. ROBERTSON: No, I cannot say off hand, Mr. Hering, but they considered that and also considered the amount of heat in the water used in water cooling and in their waste gas. They did it very thoroughly.

MR. HERING: You haven't the power factor?

MR. ROBERTSON: I have the data of that at my office.

MR. SIMPSON: I am informed that the power factor varied from 53 to 90 percent, but it is only fair to say that the low power factor was due to the breaking down of the transformer.

MR. T. D. ROBERTSON: (*Communicated*) I have received a report from Ludvika, on the working of this furnace since the new features were introduced and it may be of interest to note that the production of iron during the week September 3d to September 9th reached 3.78 tons per h.-p. year, the marked increase being attributed to the improved gas circulation.

Concerning the question of power factor raised by Mr. Hering I have obtained the following figures from the Official Report.

During January, 1911, the average tension on each phase was 62.6 volts, and the average current on each phase 14,449 amps. The average reading of the K. W. meter was 1,535 K. W., these figures giving the power factor as 0.888.

A paper read at the Twentieth General Meeting of the American Electrochemical Society, in Toronto, Canada, September 22, 1911, President W. R. Whitney in the Chair.

THE ELECTROCHEMICAL INDUSTRIES OF NORWAY.

By Joseph W. Richards.

It was the writer's intention to visit all of the electrochemical plants in Norway and to meet personally the gentlemen in charge of them, but circumstances beyond his control prevented visits to the nitrate works at Rjukan and Nottoden, the pig-iron plant at Tinfoss, the zinc works at Hafslund, the alloy plant at Trondhjem and the carbide works at Meraker. However, in the case of the last two plants, the engineers in charge have kindly furnished some information. All the other Norwegian plants have been visited, their managing directors' acquaintance made, and such a description of the companies and their plants furnished as was permissible.

Nitric Acid, Nitrates, Nitrites.

The fixation of atmospheric nitrogen had its commercial inception in Norway, and the industry has there made tremendous strides. The Norwegian Saltpeter Company, with its headquarters in Christiania, has a capital of $15,000,000 mostly of Swedish and German origin, and operates plants at Christianssand, Nottoden and Rjukan, while another large water-power is being developed for their use at Lake Tinne in the Jotunheim.

Nottoden, Telemarken.

The plant at Nottoden has been fully described in these Transactions, **16**, 131 (1909), by Dr. Schönherr. A repetition here is therefore unnecessary.

Rjukan fos, Telemarken.

This is an extension of the Nottoden plant, with 180,000 available horse-power. We understand that the large furnace units to be put up in this plant, up to 10,000 horse-power, will be on the Birkeland-Eyde principle, this having been shown to be some-

what more efficient for very large units than the Schönherr furnaces. The plant will be in operation early in 1912.

Christianssand.

The Christianssand Elektrokemiske Aktieselskabet is a branch of the Norwegian Saltpeter Company, using the Badische Anilin Company's process for making nitric acid from air. The works is on Christianssand Harbor about four miles south of the town. It takes power from the Christianssands Fossefos and Elektricitaetsverk, transmitted 20 kilometers (12 miles) from Vergeland. The plant is mostly experimental, but produces 300 to 400 tons of sodium nitrite yearly, which is sent to Germany for use in the chemical industries. This product pays better, at present, than nitric acid or "nitro-lim." The plant is under the direction of Dr. Hessberger, with Dr. Brode as research manager.

Sodium.

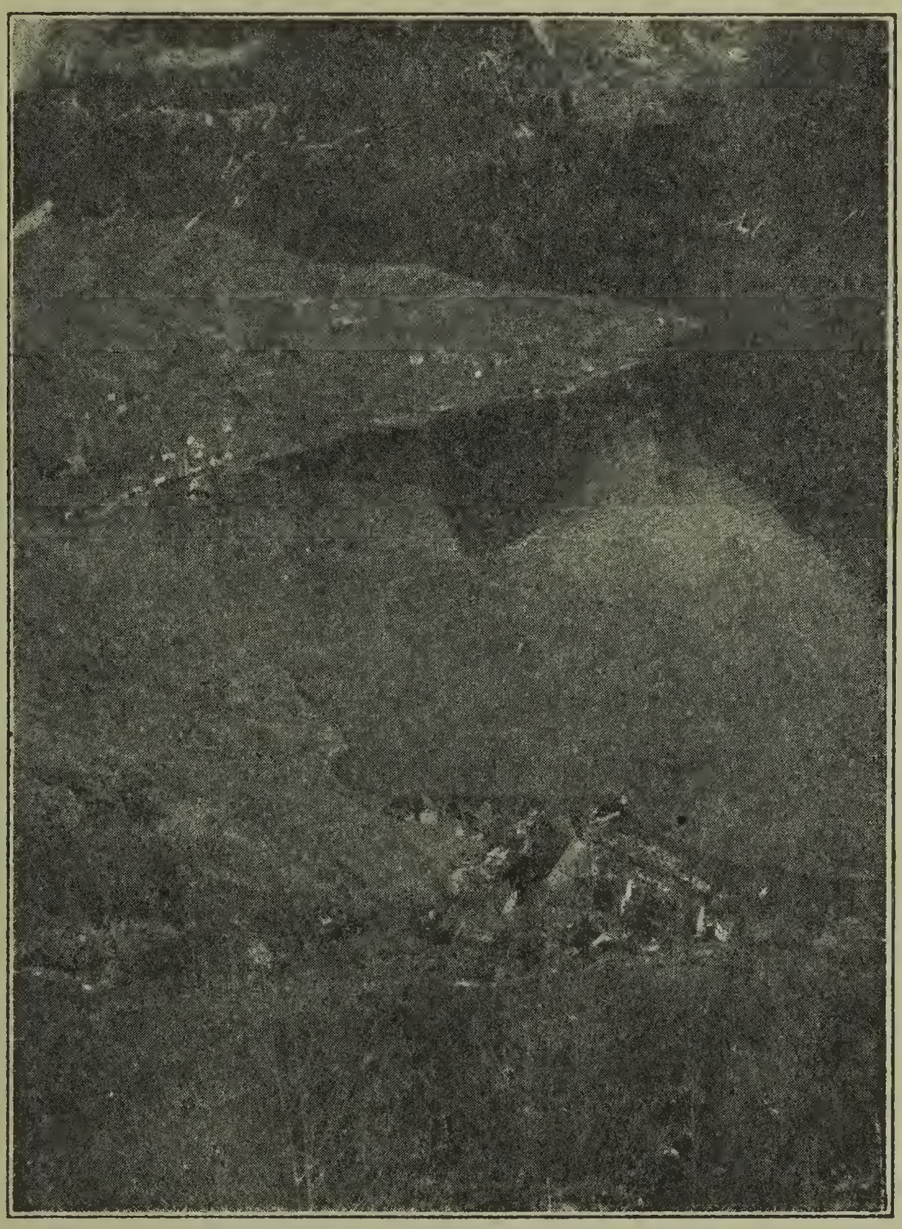
Vadeim.

The Aktien Selskab *Vadeim* Elektrokemiske Fabbriker has a plant for manufacturing metallic sodium at the head of the

VADEIM. SODIUM WORKS.

Vadeim Fjord, an arm of the great Sogne Fjord, about 100 miles from Bergen.

The works is directly on the water's edge, with a landing place for deep-sea steamers. A stream flowing here into the fjord drains 37 square kilometers (14 sq. miles), embracing several lakes at about 300 meters (1,000 ft.) above sea level. With a



VADEIM. SODIUM WORKS.

rain-fall of 1.5 meters (60 inches) this gives a theoretical power supply of 8,000 h. p. gross. At present the company uses only 450 h. p., taking 30 meters (100 feet) fall; it is, however, driving a tunnel 100 meters (325 feet) long, at an elevation of 300 meters, which will enable it to obtain 1,000 h. p. continuously, even in periods of low water. This power will be available in 1912, and

in 1913 may be enlarged to 2,200 h. p., or one-quarter of the whole power supply at Vadeim. About 1,200 h. p., of this, will be utilized for sodium. The other three-quarters belongs to Mr. E. A. Ashcroft, and may find utilization later for other electrochemical industries.

The sodium manufacture is carried on by the Castner process. The caustic soda used comes mostly from Germany; the sodium produced is shipped mostly to Glasgow or England, where it is made into cyanide. The present output is 140 to 160 tons per year. The ampere efficiency of the process averages 92 percent. The market selling price of sodium in Europe is 8½ to 9 pence (17 to 18 cents) per pound.

The plant was started by E. A. Ashcroft, was subsequently managed by I. J. Moltkehansen, and is now under the superintendence of T. L. Kolkin, a Norwegian who was once in the aluminium business, at Newcastle, England.

Aluminium.

Vennisla.

The Aktieselskabet Vigelands-Brug, otherwise The Anglo-Norwegian Aluminium Company, has its works at Vennisla, 15 kilometers (9 miles) from Christianssand, in the Saeterdal. Mr. Chateau is the managing director.

The works was started in operation in July, 1909, produced over 800 tons of aluminium in 1910 and will produce 1,000 tons in 1911. The power utilized is 12,000 h. p., which can be increased to 15,000. The fall is 15 meters (50 feet). The plant employs 230 workmen. Details of the reduction operation are not available, except that the Heroult process is used, with 90 pots, giving about 130 h. p. per pot.

The aluminium produced is 99.1 to 99.3 percent pure, and is shipped mostly to Germany and the United States.

Stangfjord.

The British Aluminium Company organized in 1906 a Norwegian company called the Aktien Selskab Stangfjorden Elektrokemiske Fabbriker, with a capital of 250,000 kronor ($67,500). This company has acquired a chain of lakes at the head of the Stangfjord, an arm of the sea about 100 miles north of Bergen and at

the most westerly point of the Norwegian mainland. The watershed is about 27 square kilometers (10 sq. miles), the rain-fall is heavy, about 2 meters (80 inches) per year, and the fall now utilized 90 meters (300 feet). The works has to shut down in March and August because of scarcity of water, but is considering diverting another stream to its use and thus enlarging its water supply to 2,500 h. p. the year round.

The works is conveniently situated close to a deep water land-

VENNISLA. ALUMINIUM WORKS.

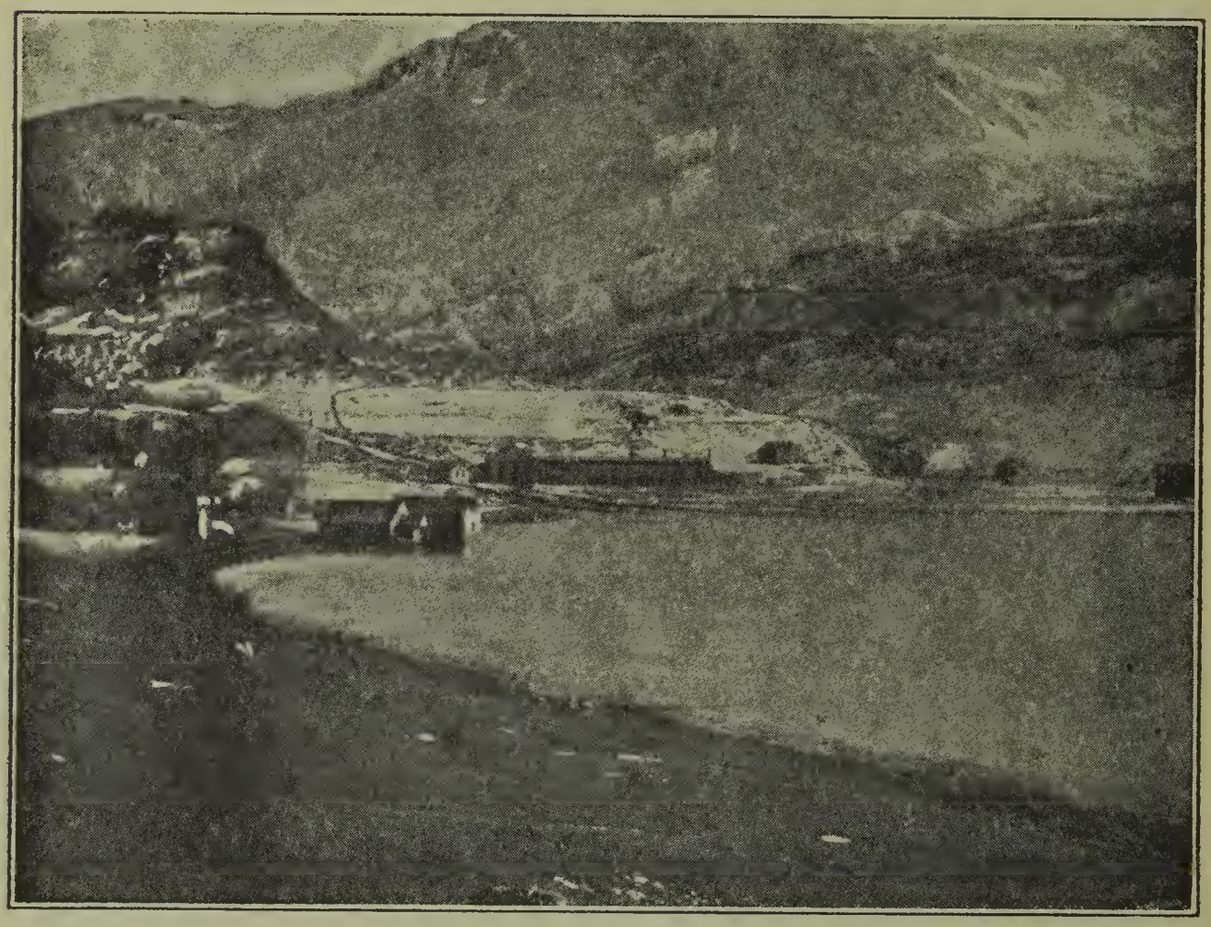
ing-place, with all the space necessary for store-houses, workmen's houses, etc. It is surrounded by mountains 600 meters (2,000 feet) high.

The process used is Mr. Heroult's. The details of the plant are not allowed to be published, except as follows: There are two direct current dynamos of 250 to 275 volts, giving each 4,000 amperes, and therefore each of 1,000 kw. The plant began producing on a small scale early in 1908. The total production to

date has been about 1,000 tons of aluminium. The market price is about 15 cents per pound.

With 2,000 kw., the average aluminium works in Europe produces 500 tons of metal per twelve months. If the plant were operated only ten months per year, its output would be 400 tons yearly.

The constructors and early managers of the plant were Mr. Blennerhasset and Mr. Skelton. The present manager is Mr.



STANGFJORD. ALUMINIUM WORKS.

R. Turner, M. Sc., a former pupil of Dr. Hutton at Manchester University. A general view of the plant is given.

Calcium Carbide.

Odde, Hardanger.

The Alby United Carbide Factories, Ltd., has a large plant at Odde. The "Alby" company has a capital of £400,000 ($1,950,000).

At Odde, 20,000 h. p., is used, transmitted 7 kilometers (4

ODDE. GENERAL VIEW. WORKMEN'S HOUSES ON TERMINAL MORRAINE, TO LEFT.

ODDE. CALCIUM CARBIDE WORKS. CYANAMIDE WORKS NEAR TOWN, LEFT OF CENTER.

miles) from Tyssaa at 12,000 volts. The cost of this power, at Odde, is 30 kroner ($8.10) per h. p. year. The annual product is 30,000 tons of carbide, of which 10,000 tons (particularly the fines) are furnished to the United Cyanamide Works immediately adjoining. The output will be doubled in 1912, the increased

output being entirely for the cyanamide works (for ammonium sulphate).

The carbide works is under the direction of Dr. A. Petersen, with Mr. Bruce mechanical engineer. The limestone used comes from near Bergen, and is burnt in vertical kilns on the premises; the coal used is English anthracite. The electrodes are supplied by Schiff (Austria). Three electrodes 30 cm. square (12 inches) are used to a 1,500 h. p. furnace. There are 12 furnaces of 1,500 h. p. each, and one experimental furnace of 2,000 h. p. The carbide is tapped every hour, about 400 kg. from each 1,500-h. p. furnace, a total of 9 tons per day per furnace.

A noteworthy feature of the furnace installation is the use of screens of double wire mesh to protect the laborers from the great heat radiated; being translucent they allow the furnace to be clearly seen.

Meraker.

The Aktien Selskab Meraker Elektriske Kraft-og Schmeltevaerk, situated at Kopperaaen near Trondhjem, Norway, manufactures calcium carbide and ferro-chromium.

The river Kopperaaen is fed by two regulated lakes, having a total area of 16 square kilometers (6.5 sq. miles), with an effective fall of 258 meters. With the minimum flow of 4.5 cubic meters per second this represents 10,000 horse-power; with the maximum flow of 6 cubic meters, 13,000 h. p. Only the lower part of the river is at present utilized, the power station having two turbines of 1,500 h. p. each, running two generators of 925 kw. each and two dynamos of 70 kw. each. The company receives a further 1,000 h. p. from the Turifos power station situated 3 kilometers (2 miles) further down the river, and later on expects to take another 1,000 h. p. from the same source. At present, the Meraker Company is therefore using 4,000 h. p., half for calcium carbide and half for ferro-chromium.

The furnace room contains two 1,000-h. p. furnaces for calcium carbide. They are a combination of the "Alby" type, enclosed in a particular way so as to work entirely closed. The annual production is 3,500 tons of calcium carbide, sold under the regulations of the Acetylene Association. The limestone comes from quarries near the Trondhjeim fjord.

The factory was burned down in 1905. It has been thoroly

modernized while being re-built. The buildings are: Power station, furnace house, carbide crushing and packing house, storage houses, work-shops, continuous lime-kiln, laboratory, offices. Since the formation of the Carbide Syndicate the plant has, in common with other works, limited its carbide output, and, thus forced to use its surplus power for other industries, took up ferro-chromium. The whole plant employs 100 workmen.

Nickel.

Christianssand.

The Christianssands Nikkelraffinierungsverke with a plant on the harbor about two miles west of Christianssand, is a development from the Evje Nikkelverk. The nickel-copper mines at Evje, 70 kilometers (40 miles) north of Christianssand in the Saeterdal have been worked for over a century, but were until recently abandoned. They are now re-opened, and the ore, carrying about 1.8 percent of nickel and 1.2 percent of copper is smelted on the spot to matte carrying 10 percent of nickel and copper. Water jacketed furnaces are used, about 8 percent of coke on the charge, and 120 tons of ore is smelted per day to 36 tons of matte.

The matte is shipped by the narrow-gauge Saeterdal railway to the works at Christianssand. Here it is first roasted dead, and then smelted in a low cupola to impure nickel-copper alloy, which is cast directly into anodes of the size usual in copper refineries. These are then treated electrolytically by Hybinette's method, the details of which are kept secret.

The nickel comes from the electrolytic refinery as deposit about 5 mm. thick upon lead cathodes. It is stripped off, and then cut into diamond-shaped lozenges, which are packed directly into barrels holding 200 kg. (440 pounds). The poorer deposits are melted down and granulated in water to supply the demand for granulated nickel. The product is sold entirely to Frankfort, Germany.

The power used is supplied by the Christianssand Fossefos & Elektricitätsverk, at 10,000 volts, transmitted 15 kilometers from Vergeland. It is sold to the company at 45 kroner ($12) per h. p.-year. The refining works transforms it to direct current of 220 volts by a motor-generator, with 15 percent loss.

Zinc.

Hafslund.

There is a considerable plant manufacturing zinc in electric furnaces, in what is described as modified De Laval furnaces, at Hafslund, near the water-fall of Sarpsborg, but the writer was unable to visit this place, or to obtain any reliable details as to the plant or methods employed.

Tyssaa, Tyssedal, Hardanger.

The Aktieselskab Tyssaafaldene, is a Norwegian company with mostly Swedish capital. Capital stock 2,500,000 kroner

TYSSAA POWER HOUSE.

($675,000). Their power plant is on the bank of the Sörfjord (Hardanger) 7 kilometers (4 miles) north of Odde. The power supply is furnished by a large lake in a hollow of the mountains, the Ringedalsvand, 450 meters (1,400 feet) above sea-level. At present 100,000 h. p. is available, which can be increased to 125,000 h. p. by regulating the lake. A granite dam 25 meters high is being built, which will raise the level of the lake 25 meters, and allow a total regulation of 50 meters. Just below the lake a tunnel through the mountain avoids the deep ravine and leads the water 3 kilometers (2 miles) to a point on the

mountain side 400 meters (1,300 feet) above the power house. Two large iron pipes descend thence at an angle of nearly 45 degrees to the power house. (See photographs.)

At the present time this company furnishes 25,000 h. p. to Odde, to be doubled in 1912, and will soon be furnishing 10,000 h. p. to the Hardanger Steel Works, at Tyssaa. The power company itself is experimenting with a 400 h. p. zinc furnace, and is also attempting the electrical manufacture of silico-manganese. It is intended to erect an electrical zinc plant, to take 20,000 h. p. Mixed sulphide concentrates will be imported from Australia, the sulphur roasted out and converted into sulphuric acid (30,000 tons per year, for the cyanamide works at Odde, for ammonium sulphate) and the roasted zinc ore smelted electrically to spelter in modified De Laval furnaces. Mr. Knut Tillberg of Westervik, Sweden, is the leading spirit in these enterprises, particularly on the financial side.

Iron and Steel.

Jössingfjord.

The Jössingfjord Manufacturing Company has a Hiorth induction electric furnace of 4-5 tons capacity, in which it manufactures high-quality steel ingots by melting together fine Swedish pig-iron and bars, as already described in these Transactions, **18** (1910). This company is impressed with the field open in Norway for electrically refining cheap steel scrap to high-quality steel, and is now erecting alongside the induction furnace a Söderberg 3-phase electrode furnace, in which the refining can be satisfactorily carried out.

The scrap comes from the Norwegian coast, also is imported, and consists of approximately half wrought iron and half soft steel, with very little cast iron. It costs at present 52.50 kroner ($14.17) per ton, delivered, but it is not unlikely that its price may rise to 60 kroner ($14.45 per short ton) in the near future.

The Söderberg furnace is circular, and can hold 2.5 tons of metal. It will have three electrodes passing through the roof, connected to a 3-phase generator by the "star" connection. Six small 2 cm. (¾ inch) electrodes embedded in the hearth will serve as connection to the bath, which latter acts as the neutral point of the system. Thus, very little or no current passes

through these embedded electrodes, while all the arcs from the upper electrodes pass in one direction only, to the bath, and are not in series with each other.

A 450 kw. generator is being installed to run this furnace, which it was hoped to have in operation in September, 1911.

Jörpeland.

The Stavanger Skibs-Ophugnings Co. A/S, is a company engaged in purchasing old ships, particularly war-ships, and breaking them up. At the present time it disposes of 3,000 to 6,000 tons of scrap iron and steel annually, which is all sold to iron and steel works out of the country, at a comparatively low price.

The managers of this company, Messrs. Poulsson, Mousen and Rugland have organized the Stavanger Elektro-staalverk Aktieselskab, with a capital of 450,000 kroner ($121,500), to utilize this scrap material in the country, by converting it into electric furnace steel, in rolled shapes and castings. The stockholders are entirely Norwegian.

The works is being erected 18 kilometers (11 miles) east of Stavanger, near a power supply at Jörpeland. The power is furnished by the Ryfylke Kraftanlaeg, which has available, at this point 10,000 h. p. The power company has a concession for eighty years and has contracted to furnish to the steel company 2,500 h. p. The first 1,500 h. p. installed will be sold to the steel company at 25 kroner ($6.75) per h. p.-year, and further power is to be supplied at 20 kroner ($5.40). These figures are for power on the turbine shaft, that is, the steel company must install and care for the electrical machinery; the power paid for is measured by the electrical power generated.

The electrical machinery will include, at the start, one monophase generator of 1,150 kv-amp., furnishing current of 25 periods to a Roeching-Rodenhauser electric steel furnace, with cos. φ 0.4 (power consumed therefore 460 kw.), and one 3-phase generator of 1,000 kv.-amp., furnishing current at 50 periods to motors for hammers, rolls, cranes, etc., where with cos. φ 0.8 the power consumed will be 800 kw.

The steel plant will contain a Siemens-Martin regenerative gas-furnace of 4 to 5 tons capacity, the producer gas being

made from English coal costing 14 kroner ($3.78) per ton. The steel scrap from breaking up ships at Stavanger, together with some English pig-iron, will be melted in this furnace and refined in the ordinary manner. When partly finished, the liquid steel will be transferred into a 3 to 4 ton Roechling-Rodenhauser induction electric furnace, of the mono-phase pattern, for further refining and finishing. This furnace uses 450 kw., at 3,000 volts primary tension, with cos. $\varphi = 0.4$.

Working these two furnaces together, day and night, with 2 hours treatment in each, it is expected to turn out 6,000 tons per year. Part of this, say 600 tons, may be cast directly into steel castings, about 1,400 tons rolled into shapes, the rest will be sold in ingot form, probably exported.

The first building of the steel works will be finished by the end of 1911, and it is expected to be making steel in April, 1912.

Mr. Fredrick Jacobsen, of Stavanger, is the electrical engineer of the enterprise, and Mr. Svenson (formerly of Dommeldingen, Luxembourg) the metallurgist. The latter has a strong preference for the monophase induction furnace, over the 3-phase, and has no doubts of the suitableness of this furnace for operating the projected refining operations.

If further extensions of this enterprise are called for, the Lyssenfjord close by has 100,000 h. p. available, which can be developed quite as cheaply as the power at Jörpeland.

Arendal.

The Aktie Selskab Arendals Fossekompagni has a total capital stock of 2,200,000 kroner ($600,000), and was organized to use the larger waterfall of Boilefos for the electric furnace production of iron and steel. The Boilefos is about 13 kilometers (8 miles) from Arendal; by regulating the lakes feeding it a continuous supply of 50 cubic meters (50 tons) of water per second can be obtained, giving 30,000 electric horse-power. This is now in course of being built. The Flatenfos, nine kilometers above Boilefos, is also available to supply 6,000 h. p. more when fully regulated.

The power will be transmitted at high tension to the iron and steel works a distance of 14 kilometers (8.5 miles). There will be installed three 3,500-h. p. pig iron furnaces of the

Swedish Ludvika Elektrometall type, and two electrode steel furnaces of 1,000-h. p. each, designed by the same company. The three pig-iron furnaces, with transformers, conveyors and building, are estimated to cost 400,000 kroner ($100,000); the two steel furnaces, with building and all appurtenances, 175,000 kroner ($45,000). The license for the five furnaces is stated as 73,000 kroner ($19,000). The plant is expected to be in operation early in 1913; at present, work is going on at the power plant and station, upon which 1,800,000 kroner ($460,000) will be expended. If to this is added 1,200,000 kroner ($325,000) to be expended on the iron and steel works, the total outlay amounts to three million kroner ($810,000). The steel works will include a rolling train, capable of turning out 14,250 tons of billets per year. When the iron and steel works is enlarged to 17,000 h. p., it will furnish 33,000 tons of billets per year.

The estimated costs of running the steel works with 12,000 h. p., and producing 14,250 tons of steel billets per year, are as follows per ton of product:

	Kroner	Dollars
Cost of power	5.30	1.43
Ore, coke, flux, electrodes	42.25	11.41
Labor	6.10	1.65
Repairs, moulds, etc	6.85	1.85
Royalty	3.15	0.85
Depreciation	20.00	5.40
Supervision, office and laboratory	2.45	0.66
Administration and commissions	4.80	1.30
Freight	7.35	1.98
Rent and interest	5.90	1.59
Miscellaneous and unforeseen	3.85	1.04
Total cost of steel billets	108.00	29.16
Selling price	140.00	37.80
Profit	32.00	8.64

It is estimated that this 12,000-h. p. plant will pay 15 percent on the capital stock, and that if enlarged to 17,000 h. p. it would pay 22 percent.

Mr. S. Eyde is chairman of the board of directors, which also includes the directors of the Norske Aktienselskab vir Elektrokemisk Industri, Dr. E. Collett and Engineer C. W. Eger.

Tyssedal.

The Hardanger Elektriske Jern og Staalverk is a Norwegian company; the manager of their works is Mr. Axel Hartman, a Swede. Their plant at Tyssedal will receive power at 12,000 volts from the A. S. Tyssefaldene, thru a conduit 800 meters long, at a purchasing price of 30 kroner ($8.10) per horse-power year, measured at the power station.

The buildings under erection are on the edge of the deep Sörfjord. They comprise ore pockets and coke pockets, served by a wire-rope tramway from the loading wharf, a crushing plant, roasting plant, office and laboratory, and a large furnace building, of iron and brick, 30 x 35 meters (100 x 115 feet).

The ore used will be partly lump ore from Klodoborg, but mostly concentrates from Rösand in the Romsdal. The latter are taken from magnetite ore with about 12 percent of TiO^2. The concentrates carry about 65 percent iron, 1.8 percent TiO^2, 0.01 P and up to 0.4 S. The concentrates will be roasted to about 0.03 percent S in a Petersen shelf furnace, using the waste gases from the pig-iron furnace. About 30 tons of ore will be roasted daily.

The limestone used will be from Mosterhavn, in the Hardanger. About 10 percent of the weight of the ore will be used, making a singulo-silicate slag, $2MO.SiO^2$.

The coke will be from Durham, England, carrying about 0.6 percent sulphur.

It is expected to make both grey and white pig-iron, low in sulphur and phosphorus, and with about 0.2 percent silicon. This will be used in the plant, or sold.

The pig-iron furnace is of the Ludvika type, similar to that operated by the Jernkontoret of Sweden at Trollhättan, Sweden. The furnace now nearly erected has the following dimensions:

Diameter of hearth	3 meters	= 10 feet.
" at ring	1.5 "	= 5 "
" at boshes	2.15 "	= 7 "
Height of shaft	12 "	= 40 "

The lining is Höganäs fire-brick, except the crucible, which is lined with magnesite. There are 6 electrodes, in three sets of two each, running on a 3-phase circuit, with 600 h. p. on each, or 3,600 h. p. altogether. The electrodes are round in section,

600 mm. (24 inches) diameter, with connecting rings kept close above the furnace arch.

The electrodes will be tapped, so as to be screwed together one after the other and thus be entirely consumed, without any butts.

A cooling tower will be used to cool the escaping gases by water spray, and thus to remove from them the bulk of the water vapor which they contain. The gases are circulated under the crucible arch. It is estimated that the furnace will produce 725 cubic meters of gas per hour and that the fan will circulate 2,000 cubic meters per hour.

There is place for a second pig-iron furnace to be built, as soon as experience is gained with this first one. There will be erected in the same building two electric steel furnaces of the Ludvika electrode type, turning out plain carbon steel.

Ferro-Alloys.

Meraker.

The Aktien Selskab Meraker Elektriske Kraft-og Schmelteverk, at Kopperaaen near Trondhjem manufactures ferro-chromium. They have a 1,000-h. p. furnace which has been in continuous operation over a year. The only repairs in this time have been the making-up of a new tap-hole. The furnace works on the resistance principle, but is different from the usual design. This and a second similar furnace of 600-h. p. are producing ferro-chromium, while an electrode furnace of 400-h. p. is used for refining the ferro-alloy. The annual production is about 1,500 tons of 6 to 8 percent carbon ferro-chromium.

Tyssedal.

The Aktieselskab Tyssaafaldene, Hardanger, is experimenting on the production of silico-spiegel and silico-manganese.

DISCUSSION.

Mr. Louis Simpson: I would ask Professor Richards how they roasted their nickel ore at the mines.

Prof. J. W. Richards: The unroasted nickel ore is smelted to matte, in a smelting furnace, at Evje, by semi-pyritic smelting.

A paper presented at the Twentieth General Meeting of the American Electrochemical Society, in Toronto, Canada, September 23, 1911, President W. R. Whitney in the Chair.

ELECTROCHEMICAL AND ELECTROMETALLURGICAL DEVELOPMENTS IN CANADA.

By Saul Dushman.

See Bibliography at end of paper for meaning of abbreviations used in foot notes.—[Editor.]

The number of electrochemical plants at present in operation throughout the Dominion of Canada is not very large. The reasons for this state of affairs are not far to seek. Up to the last decade the history of the country has been practically the story of the struggles of pioneers with the difficulties and disappointments which must always confront men in a new land. The toil of the soil, no matter how fertile it may be, is sufficient to engross in itself the attention of a small and scattered community. But the discoveries, made within the last few years, of rich mineral wealth in different parts of the country, and the exploitation by three trans-continental railroads of the vast prairies of the West, have led not only to a phenomenal growth in population but also to an influx of capital from both the United Kingdom and the United States which promises well for the electrochemical future of the country.

In view of the fact that many of you are no doubt already familiar with the existing electrochemical industries of Canada, I have given a rather brief description of these, and discussed at greater length the possibilities which this country offers from the electrochemical and electrometallurgical point of view.

We shall begin at the extreme west, in the province of British Columbia. The great Cordilleran ranges, whose peaks often reach as high as 20,000 feet above the sea level, condense enormous masses of snow, which by their annual melting serve to feed many small torrents as they come rushing down to empty into the great rivers of the lower valleys. One such tributary is the Kootenay, whose length is about 350 miles and which drains an area of 9,800 square miles. (Bull. 24.) At Bonnington Falls,

which is situated 10 miles below the city of Nelson, the river has a minimum flow of 5,850 cubic feet per second, with a natural drop of about 70 feet. In the months of June and July, when the snow melts, the flow rises as high as 60,000 cubic feet per second. At this point the West Kootenay Power and Light Company has erected two plants, the older one with a capacity of 4,000 h. p. and a later one, in which two 8,000 h. p. turbines are installed. From the latter, power is transmitted at 22,000 volts to Rossland and Trail (the latter being 28 miles distant), and at 60,000 volts to Phoenix, at a distance of 79 miles, Grand Forks, 69 miles away and Greenwood which is farthest at 83 miles. "Most of this power is used in mining and smelting work and for the lighting requirements of mining towns."[1] Besides these plants, the company operates another plant on the Kettle River, about 12 miles below the town of Grand Forks, where the head of water is 156 feet.[2] The average cost of electric power throughout the area covered by the lines of the West Kootenay Power and Light Company does not exceed one-quarter the cost of steam power.[3] The result has been that throughout the whole of the boundary district steam power is a thing of the past.

The Trail Lead Refining Plant.—As already mentioned, a large portion of the power from one of the plants is sent to Trail, where the Consolidated Mining and Smelting Company of Canada operates a plant for smelting copper and lead ores from its numerous mines. The lead bullion produced by the smelting operation is cast into anodes and refined by the well-known electrolytic process of Mr. A. G. Betts. As a full description of the plant and process is readily available in numerous publications, it is unnecessary to go into details in this connection.[4] The lead anodes contain 97.9 to 98.4 percent lead, and also some antimony, arsenic, silver and gold. A solution of lead fluosilicate containing an excess of hydrofluosilicic acid is used as electrolyte; the addition of a trace of gelatine has been found by the inventor to assist materially in the formation of coherent and smooth deposits at the cathode. The anode slimes containing most of the other

[1] Pring. p. 51.
[2] Report 24, p. 223.
[3] Ibid., p. 225.
[4] Pring, p. 53, etc. A. G. Betts, Lead Refining by Electrolysis. M. de K. Thompson, Applied Electrochemistry, p. 64-6. Report 24, p. 242-3. Electrochemical and Metallurgical Engineering, passim.

metals are treated further for the recovery of copper, arsenic, antimony, and precious metals. This plant, which was the first of its kind on the continent, was installed in 1902 with a daily capacity of 50 tons of refined lead. Since that time its capacity has steadily increased, and at present it is producing approximately 100 tons per day. An idea of its output may be gathered from the following statistics: During 1909, the total production of pure lead was 41,883,614 pounds, together with 18,241 ounces of fine gold, 2,003,003 pounds of fine silver, and 51,405 pounds of copper sulphate recovered from the slimes.[5]

There are no other electrochemical industries in the province at the present time. Proceeding towards the east the greatest development in electrometallurgical and electrochemical industries has occurred in the Province of Ontario.

Calcium Carbide.—The oldest electrochemical industry in Ontario is the manufacture of calcium carbide. The Willson Carbide Co., located at Merriton, has been in operation since 1897.[6] "The company uses as raw materials lime, coke and coal. The limestone is obtained from the quarries near Port Colborne, and contains about 92 percent of calcium carbonate. The coal and coke are imported from Pennsylvania. The raw materials are first ground in rotary crushers and rolls, and then mixed in the proper proportions, and delivered by conveyors and chutes to electric furnaces. The furnaces, slightly conical in shape, are filled gradually, the charges being weighed and fed at stated intervals. Calcium carbide is formed by the fusion of the raw materials in the electric furnaces. The fusion requires a current of from 2,500 to 3,000 amperes, at a constant pressure of 75 volts. The voltage is regulated automatically by raising or lowering the electrodes. After fusion the furnace is dumped, and the unfused material re-treated. The carbide is obtained in the form of a solid fused mass, which is crushed and bolted and finally packed into steel receptacles, each containing 100 pounds." There are, in all, six electric furnaces in operation, the power for which is supplied by three power stations, on the Welland Canal, close by. The total yearly capacity is 1,200 tons of carbide. The cost of the power is said to be very low.

[5] Report 88, p. 27.
[6] Report 24, p. 415.

The Ottawa Carbide Co., which also utilizes the same process, is located on Victoria Island, in that section of Ottawa known as the Chaudière. The capacity of the plant is 3,000 to 4,000 tons per annum. The electric power is taken from the Ottawa Power Co. The lime, containing 93-96 percent CaO, is derived from Rockland, 38 miles from Ottawa, while the coke used comes entirely from Pennsylvania.[7]

The annual output of both plants for the period 1905 to 1909 has averaged approximately 2,500 tons.[8]

Ferro-Alloys.—The work of the Department of Mines, in connection with electric processes for the production of iron and steel, has had the direct effect of stimulating at least two companies to embark upon the manufacture of ferro-alloys by the electric process.

After the completion, in 1907, of the Government experiments at Sault Ste. Marie, the Lake Superior Power Co., bought the experimental plant from the Government and used it for the manufacture of ferro-nickel pig from pyrrhotite.[9] At the present time it is utilizing the furnace to produce ferro-silicon for its own consumption.[10]

The other company which is engaged in the manufacture of ferro-silicon is the Electro-Metals Co., at Welland. The company owns about 40 acres of land to the south of the town, on the east side of the Welland canal. The iron ore is imported from the United States, and silica in the form of rock or flint is brought from Frontenac or Parry Sound district.[11] The company has four furnaces of 1,000 to 1,500 h. p. each, the daily production being 5 to 8 tons. The power is obtained from the Ontario Power Co.

Cyanamide, Graphite and Silicon Carbide.—The low cost of power on this side of the Niagara River has led to the establishment of at least two industries, in each of which power consumption is the chief item of cost.

The American Cyanamid Company, located near Niagara Falls, Ont., manufactures calcium cyanamide by the process of

[7] O. B., 1901, p. 21-22.
[8] Report 88, p. 242.
[9] Report 16, p. 93-94. Pring. p. 73-74.
[10] Report 88, p. 68.
[11] O. B. 1910, p. 30. Report 88, p. 68.

Frank and Caro, the nitrogen being obtained by passing air over heated copper, which is then regenerated by natural gas. The power is obtained from the Ontario Power Co. The company commenced operation in January, 1910, with a 10,000-ton plant. This is at present producing to its full capacity. The calcium cyanamide is not sold as such, but is previously mixed with Chili saltpeter. The whole output is shipped to the United States.

The Norton Company, of Niagara Falls, N. Y., has a plant at Chippawa for the manufacture of crystolon. The latter is a trade name for carbide of silicon. The raw materials used are two different grades of coke (a metallurgical coke with 92 percent or more fixed carbon and 5 percent ash, and a petroleum coke, containing about 91 percent fixed carbon with less than 1 percent ash) a very pure silica sand, and sawdust. Two grades of product are manufactured, green and steel gray. The power is derived from the Ontario Power Co., and amounts at present to 2,000 h. p., which is received at 12,000 volts and transformed to 145 volts, after which it passes through induction regulators which buck or boost the voltage to 70 and 215.

The whole of the production, which amounts to over 5 tons per day, is shipped to the company's main works at Worcester, Mass., where it is manufactured into different abrasive articles.

The International Acheson Graphite Company has also established a small plant near Niagara Falls. Its output in 1909 was 513,436 pounds. Incidentally it may be mentioned that both Ontario and Quebec have large deposits of natural graphite. In 1909 the exports from mills in the two provinces amounted to over two million pounds.[12]

Electrolytic Alkali.—An electrolytic alkali industry was attempted about twelve years ago by the Lake Superior Power Co. Among the many subsidiary organizations initiated by this company was the Canadian Electrochemical Company for the electrolytic manufacture of caustic soda and bleaching powder. One hundred and twenty cells of the *Rhodin* mercury type were installed, each cell utilizing 1,000 amperes at 5.5 volts. The total capacity of the plant was 4.5 tons of caustic and 9 tons of bleaching powder per day. The salt was obtained from wells in the county of Huron. The work was discontinued at the time when

[12] Report 88, p. 195-197.

it became necessary to reorganize the whole Lake Superior Corporation, and has not been renewed since then. There was then, as now, no protection for caustic soda.[13]

At the present time the Canadian Salt Co., of Windsor, Ont., is erecting a plant at Sandwich for the manufacture of caustic soda electrolytically, the Gibbs cell being used. It is expected that this plant will begin operations very shortly.

The Electrolytic Refining Plant at the Ottawa Mint.—The electrolytic refining plant at the Royal Mint, Ottawa, is a recent electrochemical development in this country. For the description of this plant I am indebted to the kindness of Dr. James Bonar, Deputy Minister of the Mint.

The gold bullion (after being assayed) is melted with silver in such proportion as to form an alloy containing 40 percent gold, 56 percent silver, and 4 percent base metals. This alloy is cast into anode plates approximately 15 inches long, 3 inches wide, and ⅜ inch thick (38 x 8 x 1 cm.). Forty-four of these anodes are suspended in a tank containing dilute solution of silver nitrate and a little nitric acid, and the silver deposited out on pure silver cathodes suspended at a distance of 2½ inches (6 cm.) from the anodes. When nearly all the silver is removed, a spongy mass of gold is left retaining the original shape of the anode plate. This is removed, washed with hot water, melted and cast into wedge-shaped plates 8 inches long, 3½ inches wide, ½ inch thick at the top tapering to ¼ inch at the bottom (20 x 9 x 1.25 x 0.6 cm.). The anodes containing approximately 96 percent gold are then refined further by the Wholwill electrolytic method, a solution of gold chloride and hydrochloric acid being used as electrolyte and strips of pure gold 2½ inches wide, 10 inches long, and 15/1000 of an inch thick (6 x 25 x 0.037 cm.). as cathodes. The deposited gold is 99.90 to 99.95 percent fine and is melted into ingots of about 500 oz. (15 kg.).

The power for the deposition is obtained from a direct coupled motor and generator which is capable of supplying 225 amperes at 30 volts when the two windings are in series, and 450 amperes at 15 volts when in multiple. When fully equipped the refinery will have 10 silver and 10 gold cells producing (with ordinary working hours) 20,000 oz. (600 kg.) of fine gold a month. At

[13] O. B. 1901, p. 61-68; Pring. p. 48-50.

the present time the output is somewhat less than half that amount. Most of the gold treated in the refinery naturally comes from the Yukon.

Electrochemical Industries in the Province of Quebec.—Most of the electrochemical industries of Quebec are situated around Shawinigan Falls. This falls is situated about 85 miles east of Montreal on the St. Maurice River. A fall of about 150 feet in the waters of this river has been utilized by the Shawinigan Falls Power Co. to develop approximately 25,000 h. p., most of which is transmitted to Montreal by an aluminum line at 50,000 volts.[14] The Town of Shawinigan Falls has been laid out with a view to the erection of a model manufacturing town, and it is therefore only natural that such a point should be chosen for the location of two electrochemical industries.

The Northern Aluminum Co., a branch of the Aluminum Co., of America, was the first industry to establish itself at Shawinigan Falls. Owing to the necessity of using direct current at low voltage, "it was considered better to generate the power as direct current. The company therefore take their supply of water from one of the penstocks leading from the canal of the Shawinigan Water & Power Co., and are expected to take the capacity of another penstock."[15] The plant covers an area of about 10 acres. The process used is the well-known reduction process of Chas. M. Hall, the alumina being prepared from bauxite in the East St. Louis (Ill.) plant of the parent company. "There are 340 cells in operation, each producing, on an average, 150 pounds of aluminum, 99.4 percent fine, per day."[16] The total capacity of the works is 25 tons of aluminum per day, about 500 men being employed when the plant is running at full capacity. The output in 1909 was over 3,100 tons.[17]

The plant of the Shawinigan Carbide Co., is situated at a distance of two miles from the power house of the Shawinigan Water & Power Co. The electric furnaces operate continuously: the manufactured carbide is drawn off in pots, and the fused mass, after cooling, is broken up and granulated in mills. Eight furnaces have been installed, two of which require each 1,500

[14] Montreal Electrical Handbook, p. 121; Pring, p. 40.
[15] Montreal Electrical Handbook, p. 137.
[16] Report 24, p. 434-435.
[17] Report 88, p. 133.

h. p., and the remaining six require each 750 h. p. for their operation. The combined capacity of the furnaces is 25 tons of carbide per day, requiring a total of 7,500 h. p. About 100 men are employed.

Besides these two plants at Shawinigan, the Electro-Reduction Co., of Buckingham, manufactures phosphorus and ferrophosphorus from apatite. No details are available as to the nature of the plant.

Part II.

This completes our survey of the existing electrochemical industries in Canada. The list is not very imposing. That, however, this country is only at the threshold of an immense electrochemical development must be evident to even the least observant. The factors which up to the present have combined to retard the industrial progress of the country, "sparse population, great natural obstacles to transportation, peculiarities of geography, and our proximity to the more wealthy and attractive country to the south," all these factors are either removed or neutralized so as to exert little or no effect. The rapid growth in population combined with the discovery of enormous natural resources must lead to an unprecedented industrial development, and considering the abundant water-power facilities of the country, the greatest developments must take place in the application of electrochemical and electrometallurgical processes.

In considering the question of the location of an electrochemical industry a number of factors have to be taken into account. There is, first of all, the problem of raw materials, second that of power, and third that of transportation facilities. Other problems must undoubtedly occur in different cases, but, in general, the above are the main factors upon which the question of the location of an electrochemical industry depends.

The discussion of the possibilities which Canada offers from an electrochemical point of view must therefore fall under the following three headings:

I. Mineral Resources.

II. Water-Power Resources.

III. Transportation Facilities.

In attempting to discuss these questions, I do so with the full knowledge that they involve not only electrochemical but also purely economical and even political considerations, in all of which few are capable of expressing expert opinion. What has, indeed, stimulated me to attempt a discussion of such a complex subject, is not the feeling that I can cast a sort of inspired light upon the problems involved, but the fact that this is the first occasion on which the American Electrochemical Society has held a meeting in a distinctly Canadian city, and it is only fitting that on such an occasion attention should be drawn to the possibilities, which we believe this country offers, of exploitation by the electrochemist and metallurgist.

Mineral Resources.

The following remarks based on the Reports of the Conservation Commission, give a general survey over the mineral resources of Canada:

A mineral map of Canada shows that "the greater portion of the country is as yet unprospected, about one-third lies within the prospected area and two-thirds without, and that the mineral resources have been developed (leaving out the Yukon) practically only in the territory lying fairly near the southern boundary. Even the portions of the country represented as being within the prospected territory must not be considered more than partially explored for minerals, as new discoveries are continuously being made within the supposed prospected areas. This has been the case at Cobalt, with the iron ore deposits of the Mattagami, and with the coal at Yellowhead pass. It will, therefore, readily be seen that the unprospected area is very great, and, considering that *much of the northern area has the same promising geological formation* as some of our known developed deposits, it is reasonable to suppose that our mineral product will be greatly increased in its development.[18] In fact, wherever prospecting has been done further north, indications of valuable minerals have been found, and, by accident, great wealth has been uncovered in some cases.[19] It is not at all an exaggeration to state that nine-tenths of Canada's mineral regions are not yet explored.

[18] C-M., p. 406.
[19] C-I., p. 19.

At the present time, the mining industry of the country is second only to agriculture. The growth in this industry from 1886 to the present has been very rapid. In the former year the total mineral production was slightly over $10,000,000; in 1909 it had grown to $92,000,000 and in 1910 to over $105,000,000. In other words the increase in 1910 as compared with the previous year is greater than the total production twenty-five years ago. Of this total slightly less than half consisted of metallic products. The statistics of the individual products are given in Table I.

TABLE I.

The Mineral Production of Canada in 1910.[20]

Product.		*Quantity.*	*Value.*
METALLIC.			
Silver	Ozs.	31,983,328	$17,106,604
Nickel	Lbs.	37,271,033	11,181,310
Copper	Lbs.	56,598,074	7,209,463
Gold			10,224,910
Pig iron from Canadian ore	Tons	104,906	1,651,321
Iron Ore (exports)	Tons	114,449	324,186
Lead	Lbs.	32,987,508	1,237,032
Zinc ore and other products			235,000
		Total,	$49,169,826
NON-METALLIC.			
Coal	Tons	12,796,512	$29,811,750
Asbestos	Tons	75,678	2,458,929
Asbestic	Tons	24,707	17,629
Natural Gas			1,312,614
Gypsum	Tons	513,313	939,838
Salt	Tons	84,092	409,624
Petroleum	Bbls.	315,895	388,550
Corundum	Tons	1,870	198,680
Other non-metallic products			1,100,664
		Total,	$36,438,278
Structural materials and clay products			19,432,854
		Total mineral production,	$105,040,958

When compared with other countries it is seen that Canada ranks well among the mineral producing countries of the world.

[20] These statistics are taken (with slight changes) from Report 102, p. 7, and are subject to revision.

The total pig iron produced was 800,787 tons, but from this has been deducted 695,891 tons which was credited to imported ores.

The lead production was about 13,000,000 pounds less than in the previous year, which "was due largely to the curtailment of production by several of the important Slocan mines consequent to the destruction of railway facilities and of several mines' buildings by forest fires."

In 1908 it stood first in asbestos, first in nickel, third in chromite, third in silver, seventh in copper, eighth in gold, and tenth in coal.[21]

The per capita production of minerals has increased from $2.23 in 1886 to $12.82 in 1909.[22] This compares favorably with the per capita production of approximately $17.00 in the United States. It is more than likely that in the next few years Canada will approach the latter figure much more closely.

The bulk of the mineral production of the country is at present derived from three provinces. In 1910, the proportion of the total output produced by each of these provinces was as follows: Ontario, 40.95 percent; British Columbia and Yukon, 23.37 percent; and Novia Scotia, 13.38 percent.[23]

In the following sections different mineral resources have been treated at greater length. For a great deal of the information I am indebted to the splendid report on Minerals issued by the Conservation Commission of Canada during the past summer. I have also made free use of the various reports and bulletins issued by the Department of Mines at Ottawa, These form a veritable mine of information on Canada's resources.

Gold.—The gold production of Canada is mainly derived from the Yukon and British Columbia. The exhaustion of the richer gravels of the Yukon has led to a diminished but much steadier exploitation of the placer deposits. The production from this territory in 1910 was $4,550,000. "The future production will probably increase annually, owing to the mining being put on a more stable basis, due to the reduction of costs and the advent of large companies. Large companies are carrying on extensive operations for dredging and hydraulic mining for the purpose of working over the old tailings and large deposits of low-grade gravel."[24]

The gold production of British Columbia was $5,432,000, in 1910, most of it being from auriferous copper pyrites ores in the Nelson and Rossland districts. It is a question whether electrolytic methods of treating these ores would not be found to be more economical than the present metallurgical methods. In

[21] C-M., p. 406.
[22] Report 88, p. 9.
[23] Report 102, p. 6.
[24] C-M., p. 408.

the treatment of the auriferous gravels of the placer deposits it is likely that some process of electrolytic amalgamation might also be found to yield better results than the ordinary amalgamation methods. The remarks of E. E. Carey in his paper on Electrolytic Amalgamation of Gold Ores,[25] are worth while quoting in this connection:

"In the near future we may look for a greatly increased production of gold, due to the application of electrochemical methods in mining and milling operations; were electro-amalgamation and electro-cyanidation today in general use in other mills now in operation, the gross output of gold would be increased 25 percent. The great increase in the future supply of gold, however, will come from vast low-grade deposits and ledges which cannot now be economically mined."

That the gold resources of the Yukon and British Columbia have only been skimmed as it were, is evident from the fact that the mountain ranges of this territory in which gold deposits have been found so far from the northern extremity of the "western or Cordilleran belt which extending from South America to Alaska is recognized as one of the greatest mining regions in the world—noted principally for its wealth in gold, silver, copper, and lead." In both Mexico and the United States this mountain range has yielded about $3,000,000 per mile of its length, and it is only reasonable to expect that Canada which possesses over 1,300 miles of this range will yield enormous amounts of the precious metals in the future. The resources of placer gold in the Klondike alone have been estimated at $100,000,000. Up to the present probably not one-twentieth of this vast area has been prospected in detail.

Silver.—About 90 percent of the silver produced in Canada in 1910 was derived from the rich silver deposits of Cobalt, Ont., the other 10 percent being derived from British Columbia. Since the discovery of the Cobalt deposits in 1904, the production of Canada has grown rapidly; in 1910 this country produced 32,000,000 ozs., or 13.3 percent of the world's total production.

Fine silver is produced at Trail, B. C., as a by-product in the lead refining process. "In Ontario, ores from the Cobalt district

[25] Trans. Am. Electrochem. Soc. **19**, 127.

are at present being treated at three metallurgical works operated by the following companies:

"The Canadian Copper Co., at Copper Cliff, Ont.

"The Deloro Mining and Reduction Co., at Deloro, Ont.

"The Coniagas Reduction Co., at Thorold, Ont.

"Silver bullion of fineness varying from 850 to 998.2 is produced at the works, other products being white arsenic, and in the case of the Coniagas plant, nickel and cobalt oxides. In each case residues carrying silver, arsenic, cobalt and nickel, are either shipped to the United States or held in reserve for further refining. In 1909, about 52 percent of the total silver production was recovered in Canada as fine metal or bullion."[26]

The ores of the Cobalt district are extremely complex, and the problem of treating these ores economically is still awaiting a solution. Under present conditions one of the pressing needs for the further promoting of mining and metallurgical activity in northern Ontario is the devising of suitable methods for the treatment of the Cobalt silver ores. Only about thirty percent of the ore shipped from Cobalt in 1909 was treated in metallurgical works in Canada, most of this being high-grade silver ores, and the rest was shipped to a number of large companies operating in the United States. "With respect to the content of nickel, cobalt and arsenic in the ores, the mining companies are paid only for a small portion of the cobalt content, and nothing for the nickel and arsenic, in fact, in certain cases the latter two are penalized."[27]

Another interesting problem in connection with the cobalt-silver ores, is what to do with the cobalt. The situation is described in the Report of the Bureau of Mines of Ontario for 1910, as follows:[28]

"The cobaltic oxide trade is at present demoralized, and is likely to remain in this condition until a greatly increased use of the article enables the demand to overtake the supply. The enforced production of cobalt ore from the mines of Cobalt has resulted in a much greater quantity of ore than can be converted into oxide and marketed as such. In fact, one year's operation of the Cobalt mines will produce ore enough to meet the present

[26] Report 88, p. 107.
[27] Report 88, p. 109.
[28] O. B., 1910, p. 24.

consumption of oxide for several years. The inevitable consequence has been a very decided fall in the price of cobaltic oxide. . . . Cobalt ore cannot at present be sold, and none is being raised from any of the silver-free veins of the Cobalt camp, the entire production being of ore associated with silver. . . . The only hope of absorbing the cobalt contents of the ores which will continue to be produced in Ontario is in an enlarged demand, brought about either by the low levels to which the prices have fallen, or by new uses for the product. It is not unreasonable to expect that the former will lead to the latter."

In the solution of these problems there is undoubtedly a field for the electrochemist and electrometallurgist. Not only is there an abundance of mineral wealth in the Cobalt region itself, but even in the districts further north, deposits of the most varied nature have been unearthed. Iron, copper, molybdenum, silver, gold, and even small diamonds have been discovered in the lone land south of Hudson's Bay. The whole of the Temiskaming region appears to be a vast mineral deposit where the richest minerals of the earth have been thrown together in the past ages, in a most haphazard and extraordinary manner—so that one knows not what to expect in the future. Added to this is the fact that there are abundant water-falls throughout the whole region. The facilities for transportation are being increased as fast as the country demands them. The government of Ontario has constructed a direct line to open up the agricultural and mining districts of the north of the province and is operating it by a commission. All these considerations serve to emphasize the fact that the hopes which are entertained for the electrochemical future of northern Ontario are not without some warrant.

Copper.—The copper production of Canada is derived from the provinces of British Columbia and Ontario. The production for 1910 from the first-named province was about 36,000,000 pounds, most of this being derived from the Boundary district. The ores of this district carry a low content of copper metal, but the smallness of the copper-content is more than compensated by the fact that the probable reserves of metal in the province are enormous.

In Ontario, there are many deposits of copper around the north

shore of Georgian Bay. Some of these are being developed gradually. The great difficulty in the development of these deposits is the lack of a smelter, as the miners experience difficulty in marketing their ore under present conditions of transportation.[29] It may be noted that similar conditions obtain with regard to the exploitation of the copper deposits of the Province of Quebec.[30]

The copper production of Ontario is, however, mainly derived from the nickel-copper ores of Sudbury, which are discussed in the next section.

Of interest to the electrochemist is the fact that "no refined copper is produced in Canada, but the copper ores are mostly reduced to a matte or blister copper carrying values in the precious metals, which is then exported."[31] In 1910 the total exports of copper contained in ore, matte and blister, according to Customs Department returns were 56,964,127 pounds, valued at $5,840,553 (practically the whole of the production) and imported in the same year 30,237,106 pounds, mostly in the form of copper bar, strip, rod and plate. As was noted in a previous paragraph, the production of British Columbia alone was about 36,000,000 pounds.

Considering the facilities for the development of hydro-electric power in the latter province it is surprising, at first glance, that no copper-refining plant has as yet been established in the Boundary district, especially when it has been found possible to operate a lead refining plant on a paying basis. To refine 30,000,000 pounds per annum would require a plant with an approximate capacity of fifty tons per day—no mean size, when it is remembered that the Balbach refining plant had this capacity in 1903 and was utilizing 750 h. p.[32] There seems to be no other reason for this state of affairs than the fact that, as the history of copper refining in the United States has shown, there has never been any general tendency to establish refineries near the mines. With very few exceptions, "the refineries from the earliest times have been established in the market centers of the East. They stand between the producer of crude metal and the consumer, and

[29] Report 24, p. 313.
[30] Report 63, pp. 69-74.
[31] Report 102, p. 10.
[32] Trans. Am. Electrochem. Soc. 3, 224 (1903).

experience has shown that they are best placed at a distributing point for the territory of consumption which they are to serve."[33]

Nickel.—The discovery of the copper-nickel ores in the Sudbury district has placed Ontario in the position of being the largest producer of nickel in the world. These deposits now supply over 75 percent of the world's total. The main producers at present are the Mond Nickel Co., at Victoria Mines, and the Canadian Copper Co., at Copper Cliff. The former is an English and the latter, an American company. The ore is first roasted and then smelted and converted into a matte, containing about 45 percent nickel and 35 percent copper. The total production of this matte, in 1910, was 35,033 tons, containing 19,258,016 pounds copper and 37,271,033 pounds nickel.[34] The whole of this matte was exported to smelting works of the two companies in England and United States, while Canada imported over $23,000 worth of nickel and nickel anodes. As Dr. Haanel has expressed it,[35] "We mine the ore, smelt it into matte, and send it as such out of the country. If we want nickel or nickel steel we have to import it. . . . Not only are these deposits of little material value to the country at present as exploited, but the method practiced is exceedingly wasteful." Dr. Haanel has, in the same connection, indicated one method by which the ores might be treated so as to prevent this wholesale exportation. His scheme, in brief, consists in magnetic separation of the iron and magnetic nickel from the copper, precious metals and non-magnetic nickel compounds followed by electric smelting of the iron-nickel portion to form ferro-nickel and electrolytic treatment of the tailings as is practiced at present in dealing with the matte. "The introduction of such a process, which would treat tailings containing the copper and part of the nickel by the electrolytic process in operation at Fredericktown, Missouri, and patented by Mr. N. V. Hybinette, would be in the interests of economy. A refinery established in the Sudbury region on the plan outlined, would enable Canada to export finished products instead of the matte, as is now done."

Nickel also occurs associated with iron and sulphur as pyrrhotite, the percentage of iron averaging about 60 when the ore

[33] Ingalls, Lead and Zinc in the United States, p. 78.
[34] Report 102, p. 11.
[35] C-I., p. 68.

is pure. The experiments conducted under the direction of the Department of Mines, at Sault Ste. Marie, have demonstrated that a good ferro-nickel pig can be produced from the smelting of roasted pyrrhotite by the electrothermic process.[36]

Iron.—The iron ore resources of the country and their probable exploitation by means of electrothermic processes, have been discussed by numerous writers within the last few years. The facts which have been established are as follows:

1. Canada has immense iron ore deposits distributed at widely different points. The deposits in Northern Ontario, in the Ottawa and Gatineau districts and in Vancouver and Texada Islands, are specially noteworthy.

2. Many of these ore deposits are situated in localities where coke for smelting operations has to be imported from Pennsylvania at prohibitive costs. In fact, it is only in the extreme eastern portion of Canada, where the iron ore of Newfoundland can be worked economically with the coal deposits of Cape Breton, that an extensive iron industry has developed. In Ontario and British Columbia coke for metallurgical purposes has to be imported.

3. The deposits are, however, usually situated near some source of water-power that could be developed at low cost.

4. The experiments at Sault Ste. Marie have shown that by the electrothermic process it is possible to smelt economically not only hematite ore, but also magnetite ores, which are the chief source of iron in this country. Furthermore, owing to the high temperature at which the reactions occur in the furnace and the consequent possibility of fusing highly basic slags, ores high in sulphur as well as titaniferous ores can be readily reduced in the electric furnace. The experiments also showed that in place of the expensive coke derived from Pennsylvania, peat coke as well as mill-refuse and sawdust can be used.

While these facts have led to very optimistic views on the possibilities of developing a large Canadian iron industry by the use of electrothermic processes, other considerations have also led investors to be rather conservative of embarking on ventures in this direction.

[36] Report 16, pp. 93-95; Trans. Am. Electrochem. Soc. **5**, 233 (1904).

The most prominent fact about the iron and steel industry of the United States is its magnitude. The necessity for economy in even the smallest detail has led to colossal aggregations in which every process has been devised by the most expert minds and carried out with the most improved machinery. To develop an industry in Canada which shall compete, for even the Canadian market, with the output of such plants, must require not only "a heavy initial expenditure in prospecting, securing and developing mines, timber lands, limestone quarries, railways, shipping docks, etc., necessary to secure a constant supply of the raw material," but also an extremely heavy outlay in the establishment of the plant itself,—*"an outlay which is much heavier in proportion to output than is required for the production of any other article of commerce."*

Outside of the heavy investment necessary for economical running the nature and extent of the ore deposit itself is of prime importance. Before any development whatever can be considered, there must be a certainty that the deposit of ore is not only extensive but also suitable for metallurgical purposes. The lack of ores suitable for treatment by the blast furnace has, in fact, been one of the factors preventing the growth of an iron industry in Ontario. In this province there were smelted, during 1909, 543,000 tons of foreign (United States) ores, while 220,000 tons of domestic ores were treated.[37] Under the present conditions the Ontario smelters have to pay a maximum figure for fuel, and are therefore naturally anxious to smelt only "those ores that yield an amount of merchantable pig iron sufficient to insure the operation of the furnace upon a profitable basis. If the smelter could secure a cheaper fuel, he could afford to smelt leaner ores, but so long as he must pay between five and six dollars a ton for his coke, furnace economy requires an ore mixture yielding a maximum percentage of iron."

By the use of electrothermic processes and some method of concentrating the ores, it is quite within the limits of possibility that a much larger iron industry could be developed in Ontario. Recent government experiments on low-grade magnetite ores from different points in Ontario have demonstrated "that first-class bessemer concentrates (with one exception) can be pro-

[37] O. B. Part I, 1910, p. 29-30; also p. 154.

duced from the crude ores submitted for testing purposes, and it has been shown that all of these concentrates will form hard porous briquettes, more or less peroxidized and free from sulphur, when submitted to a process similar to the Gröndal system of briquetting."[38] These briquettes could, of course, be treated in the electric furnace, thus cutting down on the necessity for using a high-priced fuel. The combination of concentration and electric furnace appears all the more advantageous when it is considered that Ontario contains numerous sources of water power that could be developed at low cost.

Another factor which has so far hindered the development of iron smelting plants in certain localities is the lack of a sufficient market near at hand. An excellent illustration of this is furnished by the case of Vancouver and Texada Islands. These islands have abundant supplies of magnetite ore; the numerous inlets off the coast enable transportation costs to be reduced to a minimum;[39] but at the present time no iron ore is mined for two very good reasons—lack of cheap metallurgical coke, and absence of a market close at hand.

In the electric furnace it would probably be possible to utilize the coal from Vancouver Island, of which there is an abundant supply. In a recent report on the iron ores of this region,[40] Mr. Lindeman estimates that pig iron could be produced on the coast at about $16.00 per gross ton, as compared with a present cost to consumers of $22.00 to $31.00. "It would therefore appear," he writes, "that an iron industry on the coast of British Columbia should be fairly remunerative, provided that the Province has a sufficient market to support such an industry. This is, however, not the case at present, the import of pig iron during the fiscal year ending March, 1908, being only 2,282 short tons.

"With such a limited home market, an iron industry would have to find a market for its surplus product outside the province. A large and growing market is certainly offered by the Western United States, but the manufacturers of that country are protected by a customs duty of $4 per ton of pig iron. The rapid development of the western states seems to suggest that an iron industry on the Pacific Coast of the United States will soon be

[38] O. B. 1910, p.171.
[39] B. C. B., 1910, p. 24.
[40] Report 47, p. 26.

established.[41] That such an industry, protected by the high import duty of $4 per ton on pig iron would be a dangerous competitor with a British Columbia blast furnace plant, in this American market, is apparent. On the other hand, it has often been suggested that the Orient would offer a great market. This is a question of the future, but as conditions are now, it seems impossible that a British Columbia smelting furnace, working with expensive labor and fuel, could compete with other iron producers of the world in this Oriental market.

"Though a profitable iron industry does not at present seem probable, there is room for confident anticipation that, with the prospective rapid development of the province, the conditions will be more favorable in the future, and that it will then be practicable to turn to profitable account the iron ore resources on the coast of British Columbia."

For a number of years the Canadian Government has attempted to encourage the development of an iron and steel industry in this country by subsidizing the manufacture of both products.[42] More recently bounties have also been established on iron and steel manufactured by the electrothermic process. So far, however, the iron industry has not kept pace with other metallurgical developments. The total output of pig iron during 1910 was 800,797 tons valued at $11,245,630. The total production of steel ingots and castings in the same year was approximately 822,881 tons.[43] Considering the fact that during 1910 there were imported only 158,910 tons of pig iron[44] it appears as if at the present time the production of the country is almost keeping pace with the demand. With the unprecedented growth in population there must, however, come a rapid increase in the number of engineering projects throughout the whole country, and consequently a much greater demand for iron and steel. Considering the advantages offered by electrothermic processes and their special applicability to Canadian ores, there seems to be no doubt but that there is a promising future for the electric iron and steel industry in this country.

[41] As well known, an electric iron plant has actually been established in California, since the time the above was written.

[42] Report 24, pp. 310-311.

[43] Report 102, p. 13.

[44] Report of the Department of Trade and Commerce for 1910, Part I, p. 49.

Zinc.—The zinc resources of British Columbia are considerable. In 1905 the Canadian Government appointed a commission to investigate the zinc resources of the province, and their report has now been published for some years.[45]

Zinc occurs in British Columbia mainly as blende associated with argentiferous galena. While there are large deposits of 50 percent ores in the Slocan and Ainsworth mining districts,[46] there are also enormous deposits of ores which are poorer in zinc. "Until lately no account was taken at all of zinc contents in these ores, and a large amount of this very valuable metal was lost."[47]

The attention of the Department of Mines has, however, been directed towards the problem of utilizing these ores commercially, and in the future some electrometallurgical process will probably be devised for recovering both the lead and zinc in these ores.

The question of treating the zinc ores of the Kootenay by some electric process has, indeed, received considerable attention during recent years. When it is remembered that the quantity of fuel required per ton of ore is greater in zinc smelting than in any other of the common metallurgical processes (about 1.75 tons of coal per ton of ore in the best practice),[48] it is readily seen that a process which substitutes power produced at low cost for a considerable portion of the expensive fuel, must appear desirable. It is worth noting that this factor of cost of coal influenced the Canada Metal Co., Ltd., to erect its smelter at Frank, Alberta, in the Crow's Nest Coal Field, just east of the British Columbia line.

In 1909, a Snyder zinc smelter was erected at Nelson, B. C., by the Canada Zinc Co. The intention was to treat mixed argentiferous zinc-lead ores, with a view to producing lead bullion and commercial spelter in one operation. So far, however, the plant has remained practically idle.

As already mentioned, the Department of Mines at Ottawa is interested in the problem of exploiting the ores of the Kootenay district, and an investigation is now being carried on by Dr.

[45] Report 12.

[46] According to the Report of the Commission, p. 47, "15,000 tons of ore of 50 per cent. grade would be a liberal estimate for the annual productive capacity of the Slocan." The mines of the Ainsworth camp would, according to the estimate of the Commission, produce 16,000 to 30,000 tons per annum.

[47] C-I., p. 10.

[48] Report 12, p. 51.

Stansfield, at McGill University, on characteristic ores of this district.

According to a recent report,[49] Dr. Haanel has announced, "that if the preliminary research-work indicated an electric process, the smelter built at Nelson by the Canada Zinc Company would almost certainly be used to complete the work; while, if some other process were indicated, the smelter would probably be located at Nelson in any event, for convenience to power and the ores."

The same report also contains the information that two private companies are conducting experiments with electric smelting of zinc ores of the Kootenay. It is therefore to be expected that some economical process will be devised in the near future for treating the zinc ores of British Columbia.

However, the opinion of Mr. Ingalls on the future possibilities of applying electrical methods to the smelting of the zinc ores of British Columbia seems to be rather pessimistic. He writes as follows:[50]

"It is unlikely that the electric smelting of zinc ore can ever be profitably carried on in the zinc producing districts of the East and West Kootenays, B. C., because in many parts of those districts the water powers are small and irregular. . . . Where there are large water powers, as for example, at Bonnington Falls, the power generated is so valuable for mining and miscellaneous purposes, that it could not be furnished at the low figure which would be required by an electric smelting plant.

"On the coast of British Columbia, I am informed, there are many large water powers, which can be developed at so comparatively small a cost as to permit them to furnish power at a low figure. It is possible that at some future date, these may be utilized for the electric smelting of zinc and other ores. Possibly, also, water powers in eastern Canada may be similarly utilized. Any such development is, however, something of the future, and is not to be reckoned upon at the present time. Electric smelting of zinc ore must undoubtedly go through many stages of experiment before it can be pronounced a metallurgical and commercial success."

[49] B. C. B., 1910, p. 103.

[50] Report 12, pp. 129-133.

Coal.—The following table, taken from the report of the Conservation Commission,[51] gives some estimate of the coal resources of Canada:

Province	Area of Coal Land (In Sq. Miles)	Anthracite (In Millions of Tons)	Bituminous (In Millions of Tons)	Lignite (In Millions of Tons)	Total (In Millions of Tons)
Nova Scotia . . .	992	. . .	6,250	. . .	6,250
New Brunswick	112	. . .	155	. . .	155
Manitoba	48	. . .	. . .	330	330
Saskatchewan . .	7,500	. . .	. . .	20,000	20,000
Alberta	19,582	400	44,539	60,002	104,932
British Columbia .	1,123	20	38,642	314	38,976
Yukon	400	32	32	850	914
Mackenzie Dist. .	200	. . .	. . .	500	500

The coal of Nova Scotia, which is derived from the Carboniferous formation, is bituminous, of good quality, well adapted to the production of coke and excellent for domestic uses and for steam coal.

The most important coal areas of Canada are located in the western provinces, the coal being found in the Cretaceous formation. As shown in the table, these coals are mostly bituminous and lignite, the coke from which is not nearly as suitable for metallurgical purposes as Pennsylvania coke.

A noteworthy feature is the total absence of coal areas in the provinces of Quebec and Ontario. As, however, the latter of these provinces possesses large peat bogs, the attention of the Department of Mines has been directed in recent years towards the question of utilizing this source of fuel, and very favorable results have been obtained.[52]

It may be that, as a result of the experiments performed at Sault Ste. Marie, it will be possible to utilize this peat in electrometallurgical industries. In that case, Ontario iron smelters would no longer be compelled to import coke from Pennsylvania.

Miscellaneous Resources.—While in the above paragraphs the probable electrometallurgical developments in this country have been the subject of discussion, it must not be forgotten that there are opportunities in Canada for the growth of other electrochemical industries.

[51] C-M, p. 431.

[52] Report No. 71 may be consulted in this connection.

During 1910, Canada imported $682,816 worth of nitrate of soda and over half a million dollars worth of other fertilizers. It is more than likely that with the growth of the farming industry of the country there must come a time in the near future when there will be a much greater demand for artificial fertilizers, and "Seeing that this country is almost prodigally furnished by nature with water-powers, from which electric energy can be developed at reasonable rates, there is no reason why a flourishing industry in the manufacture of air-nitrates (similar to that of Norway) should not be established for supplying not only our own home market, but also the markets of the United States and the Orient."[53]

The production of different ferro-alloys also seems to offer an inviting field. There are numerous deposits of tungsten, chromium and manganese ores throughout the eastern provinces, and there is no doubt that the exploitation of some of these deposits would be found a profitable venture.

Water-power Facilities.—A discussion of the future electrochemical developments of Canada would be incomplete without some mention of its water-power facilities.

A complete survey of the total available water-power resources of this country has yet to be made. Such a summary is promised by the Conservation Commission. A very fair idea, however, of these resources may be obtained from the reports of the Hydro-Electric Commission of Ontario and from the address delivered by Mr. Coutlee, the engineer of the Ottawa Storage Company, at the first annual meeting of the Conservation Commission.[54]

In the province of British Columbia the total amount of power already developed and in process of development amounts to about 75,000 horse power. It is impossible to state what amount of power can be obtained from the numerous water-powers off the coast, but they would certainly aggregate another 50,000 horse power.

The Mackenzie Basin presents few power possibilities, but the Basin of Lake Winnipeg, with a drainage area of 350,000 square miles "promises well for water-power when an increasing popu-

[53] Report 63, p. 7.
[54] C-I., p. 152.

lation provides the demand." Lake Winnipeg itself is an enormous reservoir 700 feet above Hudson's Bay, and it flows out through the Nelson River, which has exceptional opportunities for water-power. The rivers which empty into Lake Winnipeg also offer opportunities for water-power.

In Ontario, the sources of water-power are almost innumerable. Nearly every village and town which is anywhere near a water-power has developed its own municipal light and power plant and holds forth enticing offers to the manufacturer to locate in its boundaries. A very complete summary of the water-power available from the different divisions has been given by the Hydro-Electric Commission of Ontario. From their reports, it is probably safe to estimate that the total available water-power of the province, excluding that available from Niagara Falls and the Soo rapids, is close to a million horse power.[55] The Ottawa River alone is capable of supplying over 300,000 horse power, while the numerous falls on the Gatineau can supply over 200,000 horse power.

In the province of Quebec, "the numerous tributaries of the St. Lawrence present remarkable power possibilities, as they flow in rock basins with many abrupt falls." Montreal, Quebec, Three Rivers, and other cities along the St. Lawrence, are supplied with light and power by hydro-electric plants.

New Brunswick also possesses several rivers with exceptional power possibilities. "At the present time only the St. John River has been exploited. The Grand Forks Power Company is building a plant at that place to develop 80,000 horse power. This will be used for the manufacture of pulp and for the municipal supplies of Woodstock, Fredericton and St. John."

This brief summary will probably serve to convey some idea of the numerous water-power facilities of this country. The total water-power resources of the country have been estimated at about 16,600,000 h. p. Canada is at present in the happy position of possessing in these water-powers a source of wealth which has not been monopolized to any extent, and in view of the policy adopted by the Government of Ontario as well as of other provincial legislatures, it is certain that such a national heritage

[55] This estimate has been obtained by adding the figures given in the reports for the individual water-powers.

will be entrusted to the care of none but those who will utilize it for the people under terms to be defined by the people.

Transportation Facilities.—Next to the subject of power development, that of transportation facilities is of greatest interest to the industrial chemist and metallurgist.

The history of railway development in Canada is one of the most remarkable features of Canadian history. When it is remembered that in the year of Confederation (1867) Canada possessed only 2,278 miles of railroad while at the present time it indulges in the possession of a railroad mileage which is close to 25,000; when it is remembered that the Canadian Pacific Railway was only completed in 1886, just twenty-five years ago, after the Government had granted the company 62 millions in cash and construction and 25 million acres of land, while at the present time there are in operation two other trans-continental railways; when these facts are considered well, it is apparent that the railway facilities of the country have been increased at an enormous rate during the past decade. The various branches of these three great railways penetrate forests which five years ago had rarely felt the tread of a white man, the periodical arrival and departure of trains has become an unnoticed phenomenon in towns which have risen at the command of some modern Aladdin, as it were, in regions which not more than five years ago were as far removed from civilization as Toronto is from New York.

This completes, in a sense, the discussion of the possibilities in electrochemical and electrometallurgical developments which Canada offers. While time and space have necessarily forbidden me to dilate to any extent on any one topic, I trust that some conception at least has been conveyed of the resources of this country and of the faith which a great many individuals have in its future.

We have crossed and re-crossed in magnificent leaps of 3,000 miles the vast region comprised under the name of the Dominion of Canada. From Ungava to the Yukon, from the smiling fruitland of the Niagara peninsula to the vast Cordilleran ranges there stretches a country which possesses resources and wealth such as few can conceive.

This land endowed with a mineral wealth beyond the wildest dreams of the present generation, this land upon which has been bestowed in profusion all the advantages which go towards the forming of a great commercial nation looks forward to the twentieth century as its very own. The nineteenth century has seen the growth of a wonderful nation in one-half of the North American continent, the twentieth century will see the equally remarkable rise of a twin-nation in the other half of this continent.

In this great evolution which must occur in the next decade the electrochemical engineer will be called upon to solve many problems, not the least important of which will be that of utilizing the natural resources of this country in the most economical manner possible, and upon this task he has to concentrate all his knowledge and wisest judgment. Let us hope then that he will not be found wanting at this great opportunity.

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THE MINERAL RESOURCES OF ONTARIO.

By Thos. W. Gibson, Deputy Minister of Mines.

I am to say a few words to you this afternoon on the Mineral Resources of Ontario. Any allusions which I may make to electrochemistry, or its application to Ontario minerals, will be from a layman's point of view, and in dealing with the mineral wealth of our Province I propose to do so, not only in a brief but in a popular way.

Before touching upon my topic proper, let me call attention to the size and situation of the Province of Ontario. It has an area of 220,000 square miles, say 140,000,000 acres. It is 1,000 miles in length from Mattawa to the Manitoba boundary, and has an extreme width from the north shore of Lake Erie to James Bay of about 600 miles. On the south its frontier marches with the boundaries of the States of New York, Pennsylvania, Ohio, Michigan, and Minnesota, and its western boundary is about midway between the shores of the Atlantic and Pacific. It has a sea coast 300 miles in length on James Bay, and this line will be longer, and the provincial territory larger, when the question of dividing the district of Keewatin is finally settled.

Geologically, the prevailing rock formations are old, most of them very old. The southwestern peninsula of Ontario, that portion partially enclosed between Lakes Ontario, Erie, St. Clair and Huron, and south of a line drawn say from Midland to Kingston, is occupied by Devonian and Silurian series. These formations are repeated on the slope north of the Height of Land, and form a coastal plain surrounding James Bay, with a smaller outlier on Lake Temiskaming. The remaining and much larger portion of the province, lies within the great Pre-Cambrian shield which occupies perhaps one-half of the whole Canadian territory, and whose southern fringe extends into the States of

Michigan and Minnesota. The Pre-Cambrian are pre-eminently the mineral-bearing formations of Ontario.

The stratified Silurian and Devonian rocks are by no means devoid of useful minerals. In the counties of Lambton and Kent are the petroleum wells of Ontario. Those of Lambton county have been producing oil for forty-five years. The annual production is declining and is now a little over one-third what it was fifteen years ago. The oil is found in the Corniferous limestone, where the wells are about 400 feet in depth, and in the Niagara, Clinton and Medina strata at 1,300 or 1,400 feet from the surface. A remarkable feature of the oil wells is their small daily production, which is probably not more than eight or ten gallons, and only their large number, say eight or ten thousand, serves to maintain the output even at the declining rate. The shallow wells in the Corniferous formation are easily and cheaply worked by the "jerker" system of pumps, one engine serving a dozen or more wells. The production of oil in 1910 was eleven million Imperial gallons. There is, I may say, a bounty paid by the Government of Canada of one and one-half cents per gallon on all oil raised.

If the production of petroleum is lessening, that of natural gas from the same region is rapidly increasing. The output for last year was valued at $1,500,000, the valuation being at a low rate per thousand cubic feet. There are three principal gas fields, one in Welland county, one in Haldimand and Norfolk, and another in Kent and Essex. These fields are gradually being extended, and there seems a possibility that the gas belt will ere long cover practically the whole of the north shore of Lake Erie. Natural gas forms an ideal fuel for domestic purposes. It can be turned on and off at will, it leaves no ashes and makes no dirt, and as compared with either coal or wood, it is much more economical. About 200,000 people in the southwestern peninsula enjoy the use of this fuel, and it would be in the public interests if it could be preserved for domestic use only, and not employed as well, as it is now, in manufacturing purposes, in burning lime and brick and for the generation of steam.

Salt beds of great thickness and purity underlie the eastern shores of Lakes Huron and St. Clair. The salt occurs in the Onondaga formation, and is pumped to the surface in the form

of brine; the brine is evaporated in open or vacuum pans, and the process is manipulated so as to produce grades of salt differing in the size of the crystals, according to the purposes for which it is required. The production of salt last year was about 84,000 tons.

In the valley of the Grand river are extensive deposits of gypsum, which have long been mined on a small scale, but operations are now becoming more extensive. It is used for the ordinary purposes, such as plaster of Paris, bug poisons, wall plaster, etc. The development of the manufacture of Portland cement is affording an increased outlet for gypsum, which is added in small quantities to the cement in order to regulate its setting properties.

Stone for building and road purposes is extensively quarried, also limestone for the manufacture of lime and cement, and marl for the latter.

Clay is abundant and is universally used in the manufacture of brick, tile, sewer pipe, and other products. It is a fortunate circumstance for the dwellers in southern Ontario generally that building materials, such as stone, lime, clay, and cement are plentiful and cheap. This climate requires adequate shelter, and the housing of the people in winter is no unimportant item. Visitors to Toronto, and other Ontario cities, cannot fail to notice the substantial appearance which is produced by the prevailing use of brick and stone for building purposes.

This part of the Province does not yield any workable deposits of the metallic ores. A little bog iron ore was obtained in the early days of Ontario, out of which were fashioned the stoves and potash kettles of the pioneers. Galena and zinc occur as pockets, here and there in the limestone, but as yet all such finds have been unimportant.

Eastern Ontario affords a great variety of minerals, including iron, gold, pyrites, lead, mica, graphite, talc, feldspar, corundum, zinc, quartz, molybdenite, etc. All of these are worked, and constitute the basis of important local industries.

The Huronian and Keewatin formations of northern Ontario contain the chief metallic deposits of the province, including silver, nickel, iron and gold. Of these, the two most important at present are silver and nickel.

There have been two distinct eras of silver production, one beginning a little over forty years ago at Silver Islet, a tiny island off Thunder Cape in Lake Superior, and spreading to the mainland west of Port Arthur. Over five million dollars worth of silver was taken from this region, but production practically ceased owing to the heavy fall in the prices of silver about 1896. In 1903 the first discoveries were made at Cobalt, whose mines have proved phenomenally rich and have become world-famous. Up to the end of 1910 the production from Cobalt amounted to about ninety-four million ounces, valued at a little over forty-eight millions of dollars. For the first six months of 1911, the production was upwards of fifteen million ounces valued at seven million six hundred thousand dollars. Cobalt is now in the flood-tide of her yield, and will doubtless be producing silver for years to come. This camp has made Ontario the third largest silver-producing community in the world, being outranked by Mexico and the United States only. About 15 per cent. of the world's yield of silver comes from Ontario.

Associated with the silver at Cobalt are cobalt, nickel and arsenic. These minerals, however, yield nothing to the mine owners, and consequently it is difficult to get accurate statistics of production. The principal use of cobalt is in making the beautiful color called cobalt blue, largely used in the manufacture of china, enamel-ware, etc. The enforced production of cobalt from these mines has brought about a reduction in the price of cobalt oxide and is likely to lead to still further reductions. The proportion of nickel in the ores is not large, and the total nickel output is unimportant as compared with that of Sudbury. The arsenic is recovered in the refineries as white arsenic or arsenious acid, and is used in making insecticides, paints, Paris green, and in the manufacture of glass, to which it imparts a peculiar brilliancy.

In the production of nickel, Ontario leads the world, her only rival being New Caledonia, an island in the Pacific Ocean, used by France as a penal colony. The nickel fields of Ontario are situated at Sudbury, and the ore occurs as a pyrrhotite mixed with chalcopyrite. The nickel bearing mineral is pentlandite. The ore after being raised to the surface is crushed and roasted in the open air in huge heaps in order to expel the sulphur. These

heaps vomit into the air every day hundreds of tons of sulphuric acid in the form of sulphurous fumes, not only wasting the sulphur, but blasting every green thing within miles. After being roasted, the ore is smelted in blast furnaces, and then converted into Bessemer matte, containing about 80 per cent. metallic contents, say 45 per cent. nickel and 35 per cent. copper. The matte is then exported, partly to the United States and partly to England, where the final separation takes place. The production of nickel last year amounted to 18,636 tons, worth in the matte about four million dollars, but on the basis of the refined metal at New York, worth nearly fifteen million dollars.

The copper output was 9,630 tons, valued at $1,374,000. It may be mentioned that along with the nickel and copper these ores carry small proportions of cobalt, platinum, palladium, gold and silver.

A large extent of so-called "iron formation" is found in the Keewatin rocks of northern Ontario. Iron formation is not necessarily iron ore, and all iron ores are not necessarily workable. A good deal of the iron ore found in northern Ontario is in the form of banded magnetite, and the accompanying silica in many cases reduces the metallic contents to a point at which the deposits are not commercially workable. Some of these bodies are very large, for instance those on the northeast arm of Lake Temagami, east of Lake Nipigon, and elsewhere. Moose Mountain, Atikokan and Magpie have large bodies of workable magnetic ore. A proportion of the ores will require concentration, but much is quite rich enough for immediate use. At Grand Rapids on the Mattagami river, north of the Height of Land, occurs what is apparently a large body of iron ore, associated with limestone. It is believed to have resulted from the oxidation of siderite, of which there is reason to think considerable areas occur in the same neighborhood. At Helen mine, in the Michipicoten district, a large deposit of hematite has been worked for about ten years, and now produces from three to four hundred thousand tons a year. It is remarkable for including in the ore body large pockets of granular iron pyrites of high quality.

Gold is scattered more or less sparsely over large areas in northern Ontario. Hitherto it must be confessed that the business of gold mining in this province has been unprofitable. On

Lake of the Woods, on Rainy Lake, Seine river, on the north shore of Lake Huron, at Lake Wahnipitae, in the county of Hastings, and other places, shafts have been sunk, stamp mills erected, and more or less gold produced, but the industry has never attained a permanent or profitable footing. There are now grounds for hoping that this state of affairs will come to an end. The discoveries at Porcupine were some of them phenomenal in their showings of free gold, and although the future of a gold camp is a difficult thing to forecast, we may hope that there will be a number of paying mines in that region.

There are many deposits of copper sulphide on the north shore of Lake Huron. One of these at Bruce Mines, was worked fifty years ago, and produced on the whole about three and one-half million dollars' worth of copper. The deposits are, for the most part, unworked at the present time, but if copper should rise in value, no doubt these mines will some day come into play. At present the chief source of copper in Ontario is the nickel-copper mines of Sudbury.

There are extensive bodies of iron pyrites, which are used in the making of sulphuric acid, in various parts of Ontario, east, northeast and northwest. Perhaps the largest mine at present being worked is near the junction of the Trans-continental Railway and its Fort William branch. This is called the Vermilion mine. Other mines are being worked in Hastings county, and along the line of the T. & N. O. railway.

The members of this society are better qualified to judge than I am, of the opportunities there are for the application of electrochemical methods to the treatment of any of the minerals which I have mentioned. Iron ore naturally occurs to one's mind in this connection. It can hardly be said that as yet the electro-thermic process of making pig iron from iron ore can successfully compete with the modern blast furnace, which is a pretty efficient piece of apparatus, but in the manufacture of special grades of steel the electric furnace has already won an assured place. A demonstration was given a few days ago at the Toronto Fair, by which titaniferous iron ore was converted in the electric furnace into a high quality of steel. The apparatus was designed and operated by Mr. J. W. Evans of Belleville. In the concentration of low grade magnetites electricity plays an

important part in eliminating rock matter and reducing the sulphur contents. The electric processes for producing pig iron and steel would be specially adapted to a country like Ontario where water power is abundant, and where there is no native coal. In the reduction of other metals heat generated by electricity is displacing heat produced by the oxidation of carbon, as being more convenient and economical. The electric method is in use at present at Welland, for the making of ferro-silicon, the quartz for which is brought from the Georgian Bay. For the refining of copper electrolysis has of course long been practiced, and attempts have been made to refine nickel in the same way. A solution of salt is decomposed by the electric current, and the resulting chlorine attacks the metals in the matte forming chlorides, the copper and nickel contents of which are successively removed from the solution by electrolysis. The electrolytic method has also been applied in the United States to the refining of zinc, but so far the old processes do not appear to have been displaced. In the treatment of non-metallic substances electrochemical methods have a large field. Calcium carbide, for instance, has for ten or twelve years been widely manufactured in the electric furnace, and provides an abundant supply of acetylene gas for illuminating purposes. From common salt, caustic soda and bleaching powder were formerly produced by electric methods at Sault Ste Marie, after the patents of Mr. J. G. Rhodin of Manchester, England. Electrical methods have also been applied for the production of bicalcic phosphate for use as a fertilizer from low-grade or unserviceable deposits of phosphate of lime, and are at the present time in use in Quebec for the production of phosphorus from the same mineral. The abundance of limestone—some of it nearly pure carbonate of lime, and some of it rich in magnesia, provides material for the extension of electric methods.

There are doubtless other applications of electrochemistry which will occur to members of this Society. I may be permitted to suggest for their consideration, a couple of problems, the solution of which would be highly desirable.

In this Province there are large deposits of corundum, which is nearly pure alumina, containing 53 per cent. aluminium. Attempts have been made to produce metallic aluminium from

corundum, but so far without success. It is a much richer ore than bauxite, the one usually employed, which carries about 40 percent aluminium, and if a serviceable electric method could be devised for obtaining metallic aluminium from corundum, both the inventor and the public would no doubt be benefited.

The other problem is provided by the presence of about 13 or 14 percent of potash in feldspar, of which large masses are found in certain parts of Ontario. Potash is a prime necessity for maintaining the fertility of soil, and at present the supplies of this article are entirely confined to Germany, and are under German control. Why should not some ingenious electro-chemist devise a method for dissociating the potash from feldspar, and thus win for himself, and for the agricultural interests of this and other countries, a rich reward?

A paper presented at the Twentieth General Meeting of the American Electrochemical Society, in Toronto, Canada, September 21, 1911, President W. R. Whitney in the Chair.

TRANSFORMING STATIONS OF NIAGARA ELECTROCHEMICAL AND ELECTROMETALLUGICAL INDUSTRIES

By A. J. Jones.

Grouped around Niagara Falls are seven hydro-electric generating stations, four of which are on the Canadian side. From these seven stations energy is being used at the rate of 128,000 h. p. for electrochemical purposes, 56,000 h. p. for railway service, 36,000 h. p. for lighting, and 55,000 h. p. for various industrial services, the total being 275,000 h. p., or about 5.5 percent of the available power of the cataract. Of this total amount 146,000 h. p. is employed locally in industries that have been attracted to Niagara Falls by reason of the generating stations located there, electrochemical processes forming 87 percent of this amount and 46 percent of the total amount utilized.

The brief description here given of some of the installations for transforming and distributing this power in electrochemical plants, together with a discussion of the more salient features, it is hoped, may prove of interest. In any event, the rapid progress of the electrical art makes desirable a frequent comparison of notes regarding methods of design and operation.

International Acheson Graphite Company.

The new 11,000-volt transformer station of the International Acheson Graphite Company with a capacity of 2,800 kw., made up of one 1,600 kw. and three 400 kw. units, is thoroughly modern in all the details of its design.

This station is supplied with power by The Niagara Falls Power Company. Two 3/0 (85 sq. mm.), 3-conductor, lead-sheath cables, with provision for a third cable, each capable of transmitting approximately 3,200 h. p., connect this station with the step-up transformer plant of the power company. A second station operated by the International Acheson Graphite Company

receives its energy from the same power company but in the form of 2,200 volt, 2-phase power. This station is fed by two 2-phase underground feeder systems, each system being made up of four

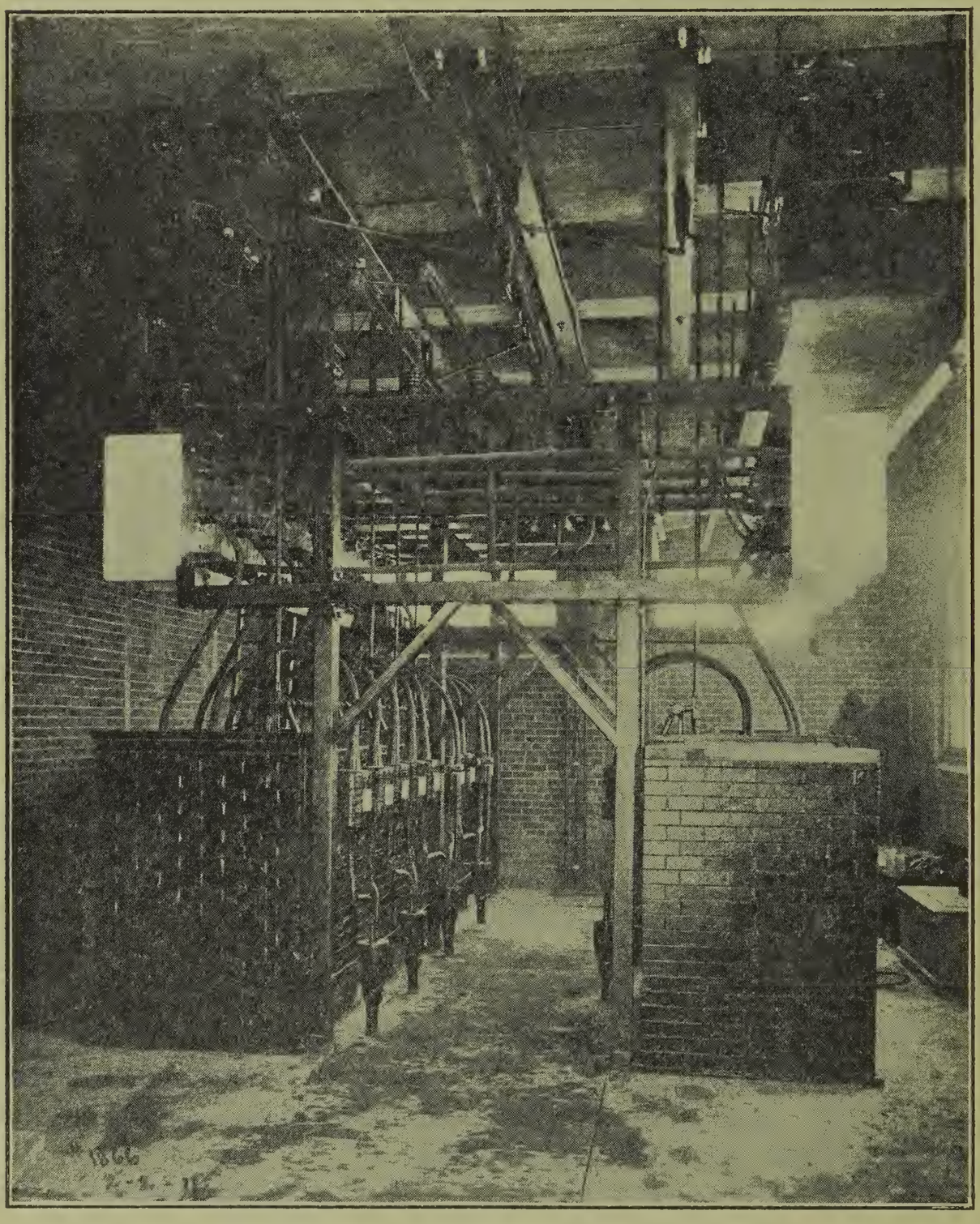

FIG. 1.

1,000,000 cir. mil. (506 sq. mm.) lead-sheathed cables with a capacity of 2,000 h. p. per phase.

The switch room of the 11,000 volt station is situated on the

west side of the International Acheson Graphite's new furnace room in a two story brick building with a floor space of approximately 21 ft. by 41 ft. (6.3 x 12.3 meters). On the lower floor are located the switching apparatus, bus bars, meter transformers, etc. (Fig. 1). On the second floor are located the switchboards and low voltage control apparatus.

The incoming power cables enter through a subway which extends the full length of the building. On each side of the subway are erected the masonry cells enclosing the oil circuit-breakers for both the incoming and outgoing circuits. Directly over the subway above the oil circuit-breaker cells is located the bus-bar structure made of seasoned maple. Disconnecting switches mounted on porcelain insulators fastened to marble slabs are bolted to each side of the bus-bar structure. Suspended from the ceiling in line with the disconnecting switches are two platforms containing meter transformers.

Each incoming power cable terminates in a brass pot head; thence passes through current transformers to the oil switch; thence to disconnecting switches to the bus bars.

The oil switches are of General Electric make, form K12, 15,000 volt, of 300 ampere capacity, operated from the switchboards above by link connection.

There are four switchboard panels of blue Vermont marble 7 ft. 6 in. (2.25 meters) high. One panel 30 in. (0.75 meter) wide controls the three incoming cables. On this panel are mounted two watt-hour meters which measures the total power delivered to the bus bars, a bracket-type voltmeter and time-limit relay of the bellows type. The other three panels are 20 in. (0.5 meter) wide and control the 1,600 kw. unit and two 400 kw. units, the third 400 kw. unit being located in a separate building with its switching apparatus in the same room with the transformer. On each of the unit panels are mounted an ammeter, indicating wattmeter, special voltmeter for indicating the position of the secondary switch, a secondary voltmeter, watt-hour meters and control switches for operation of the regulator switch.

The regulator transformers are located a short distance from the switch house in separate bays and are fed by a duplex, lead-sheathed cable. The oil circuit-breakers that control the transformers are of the same type as the circuit breakers on the incom-

ing cables except that they are 2-pole instead of 3-pole. The selector switches are single pole, double throw, so arranged that the units may be connected to any one of the three phases.

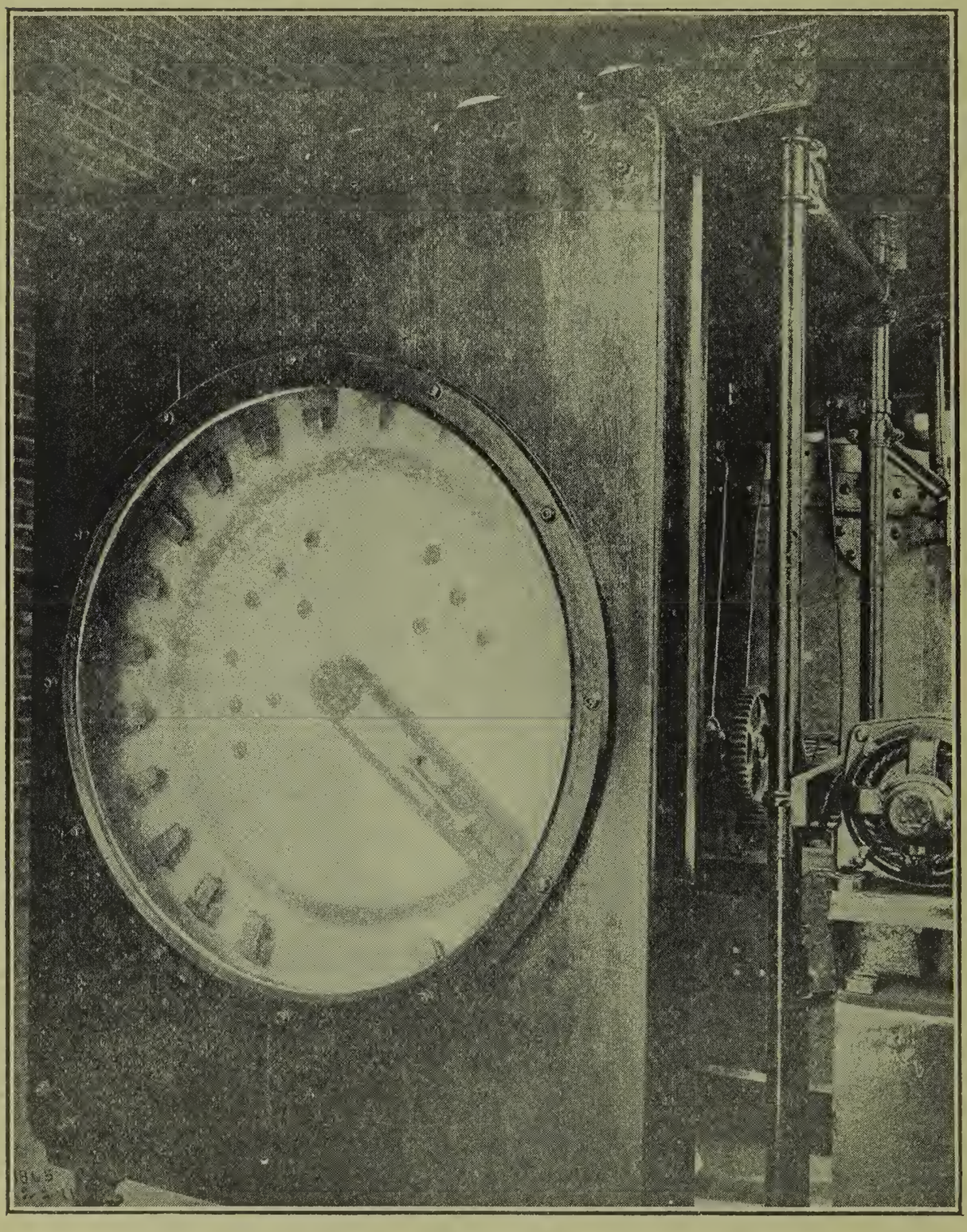

FIG. 2.

The regulators represent the latest development of the General Electric Company and are known as the transformer type of regulator. They are oil-insulated water-cooled with primaries wound

for 11,000 volts. The 1,600 kw. unit is capable of a voltage variation of 40 to 160 volts with full output at any voltage. Two of the 400 kw. units have a voltage range of 80 to 160 volts. The third 400 kw. unit has a secondary voltage variation of 30 to 60 volts. The regulation is accomplished by means of a dial switch which is rotated by an induction motor controlled from the switchboards in the switchroom. The rotating switch of the 1,600 kw. unit consists of two dials of 22 points each mounted on a 2-in. (5 cm.) marble slab in a dust proof case. The 400 kw. units have single dials of the same number of points as the 1,600 kw. unit (Fig. 2). As all the regulating is done on the primary side of the transformers; the volts per step are quite high. In order to have this switch meet the heavy duty required of it, manufacturers have arranged two electrically operated switches, or contactors, in series with each half of the rotating switch. These operating circuits are so connected to a small contact device on the main switch as to open the auxiliary contactor and interrupt the contact and again close the circuit after it has made contact with any of the various stationary contacts. With this arrangement all arcing is removed to the auxiliary contacts which are made with heavy and easily replaceable parts.

The arrangement of the 2,200 volt station is similar to the 11,000 volt station just described. The incoming feeders enter through a manhole in the lower floor of the switch house which is situated on the west side of the graphite furnace room. The incoming power passes through General Electric K4 oil circuit-breakers, thence through knife switches to bus-bars mounted on an iron framework back of the switchboard. On the main floor of the switch house, in addition to the receiving panels, there are located two 150 kw. and two 75 kw. oil-insulated, self-cooled General Electric transformers with 230 volt secondaries which supply power to the motor circuits; two 17½ kw. oil-insulated, self-cooling transformers with 110 volt secondaries used for lighting, two water switches for control of the main units, one 180 kw. induction regulator and one 5-kw. motor generator set used for arc lighting and experimental work. On the second floor of the switch house are located three switchboard panels controlling the motor and light transformers and main regulating transformer, also three low-tension panels connected to the low tension

side of the motor and light transformers. The high tension panels are constructed of blue Vermont marble 24 in. (0.6 meter) wide mounted with oil circuit-breakers, ammeters, volt meters,

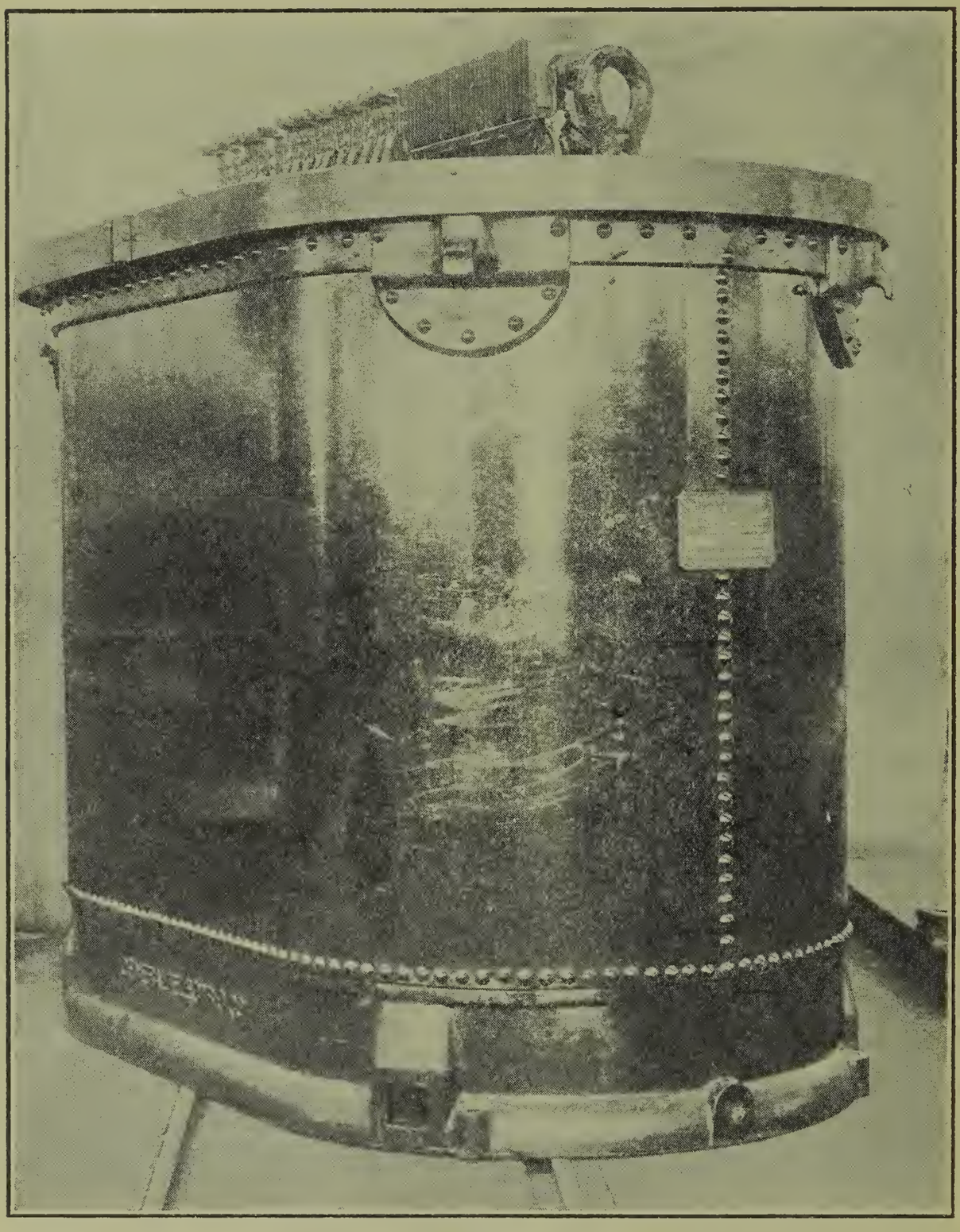

Fig. 3.

and watt-hour meters. The low tension panels are equipped with knife-switches, cartridge-fuses, and 3-pole, 600-volt, carbon-break circuit-breakers.

There are two regulating transformers used for furnace operation, each with a capacity of 1,600 kw., one manufactured by the Westinghouse Electric & Manufacturing Company and the other by the General Electric Company. They are oil-insulated water-cooled. The Westinghouse unit is capable of a voltage regulation of 80 to 190 volts and is made up of a 180 kw. induction regulator in series with the primary of the main auto-transformer. Taps of the main transformer are connected to a series of oil switches which are opened and closed by drum and cam arrangement operated by a small induction motor controlled from the switch room. The main transformer gives a step by step regulation, the regulator being used to vary the voltage gradually between each step on the main transformer.

The General Electric unit (Fig. 3) is similar to the 11,000 volt units just described except that it has an additional drum-switch and is capable of giving a straight line voltage variation of 40 to 200 volts, part of this being accomplished by a motor operated knife-switch in the secondary (Fig. 4). The accompany diagram (Fig. 5) shows the connections of the dial-switch and drum-switch (Fig. 6). The two regulating units are housed in a small brick building in the center of the furnace room. The low tension bus-bars are constructed of aluminum and extend the full length of the furnace room.

The Aluminum Company of America.

Electrical energy is supplied the Aluminum Company of America at its No. 1 Niagara Works by The Niagara Falls Power Company, delivery being made at three stations known as 1A, 6A, and 10A. These stations are designated by the number of the buildings in which they are located and are of two classes, one containing rotary converters, the other regulating transformers.

The rotary converter station is of interest as being the first station to receive power from The Niagara Falls Power Company. Power was first delivered to this station in August, 1895, since which time it has been in continuous operation with very few interruptions. The equipment consists of 600 kw. General Electric rotary converters, 2-phase, 25-cycle, running at 187.5 r. p. m., some of which are located on the west side of the station and

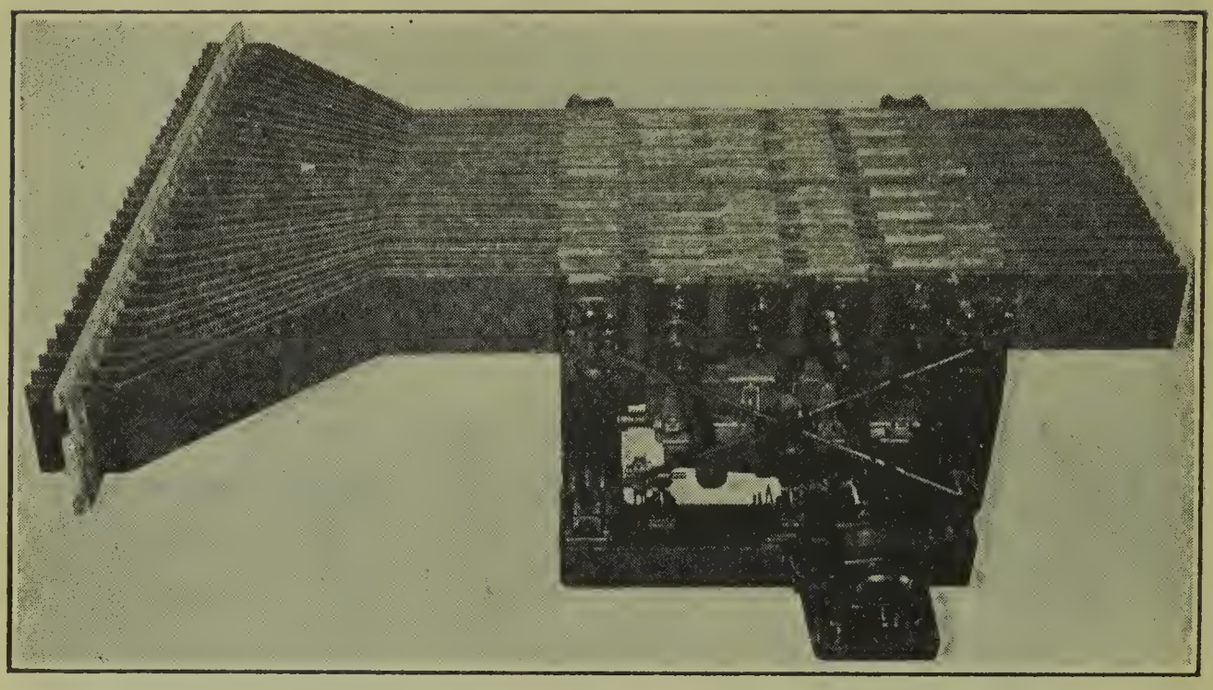

FIG. 4.

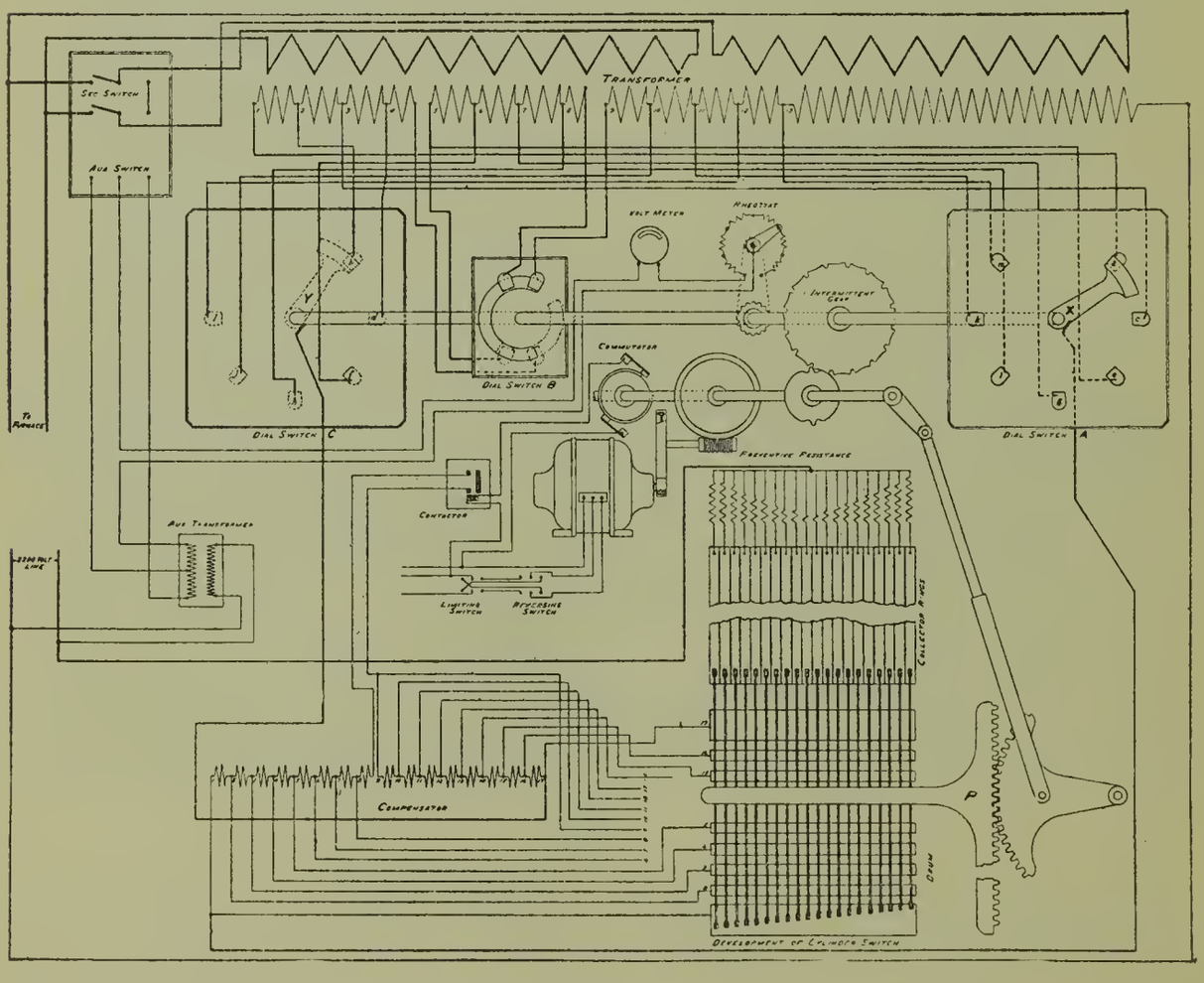
TRANSFORMER
SEC. SWITCH
AUX. SWITCH
VOLT METER
RHEOSTAT
INTERMITTENT CELL
COMMUTATOR
DIAL SWITCH B
DIAL SWITCH C
DIAL SWITCH A
TO MOTORS
CONTACTOR
PROTECTIVE RESISTANCE
AUX. TRANSFORMER
2200 VOLT LINE
LIGHTING SWITCH
RECORDING SWITCH
COLLECTOR RINGS
COMPENSATOR
DRUM
DEVELOPMENT OF CYLINDER SWITCH

FIG. 5.

others on the east side, the middle section of the station being occupied by banks of air-blast, step-down transformers. The transformers are single-phase with 2,200 volt primary, and are supplied with air by two blowers driven by direct-current shunt motors located on the floor of the station. A subway is carried under the transformer which conducts the air from the blowers to the transformers.

Alongside of each bank of transformers and in line with the rotary shaft is a switchboard panel which controls the primary and secondary circuit of each bank. This panel is equipped with four 2,000-volt knife-switches, without barriers, mounted at the top of the panel and four low-voltage knife-switches mounted at the bottom.

The direct current side of the rotary is controlled by a panel provided with knife-switches, field-switch and field-rheostat handle. All of the rotaries are connected on the direct current side to a bus-bar, supported on an iron framework on the west side of the station, the leads from the two rotaries on the east side being carried under the floor in a subway.

In the north end of the station are the receiving panels, two in number, one of which is equipped with single-throw, single-pole knife-switches, the other with two automatic double-pole, 1,500 ampere, type B, Westinghouse oil circuit-breakers. These panels are made of asbestine stone and are mounted side by side on an angle framework. The incoming power cables enter through a subway and manhole under the receiving panel. They are single conductor, lead-sheathed, with a cross section of 1,000,000 cir. mils. (506 sq. mm.) each.

Transformer station 6A is located in a two-story brick building tacked on to the south wall of the carbon furnace plant. In this building are located the switchboards and control switches for six regulating transformers located in transformer houses in the furnace room. On the ground floor is an asbestine stone panel to which are connected six 1,250,000 cir. mil. (632 sq. mm.) single conductor, paper and lead cables constituting a 2-phase and a single-phase feeder system. From the disconnecting panel the cables pass up to a marble panel on the second floor, mounted with two double-pole, remote-control, automatic, type B, Westinghouse oil circuit-breakers and necessary meters. A two-phase bus-bar

system in the rear of the switchboard receives the current after it passes through the oil circuit-breakers.

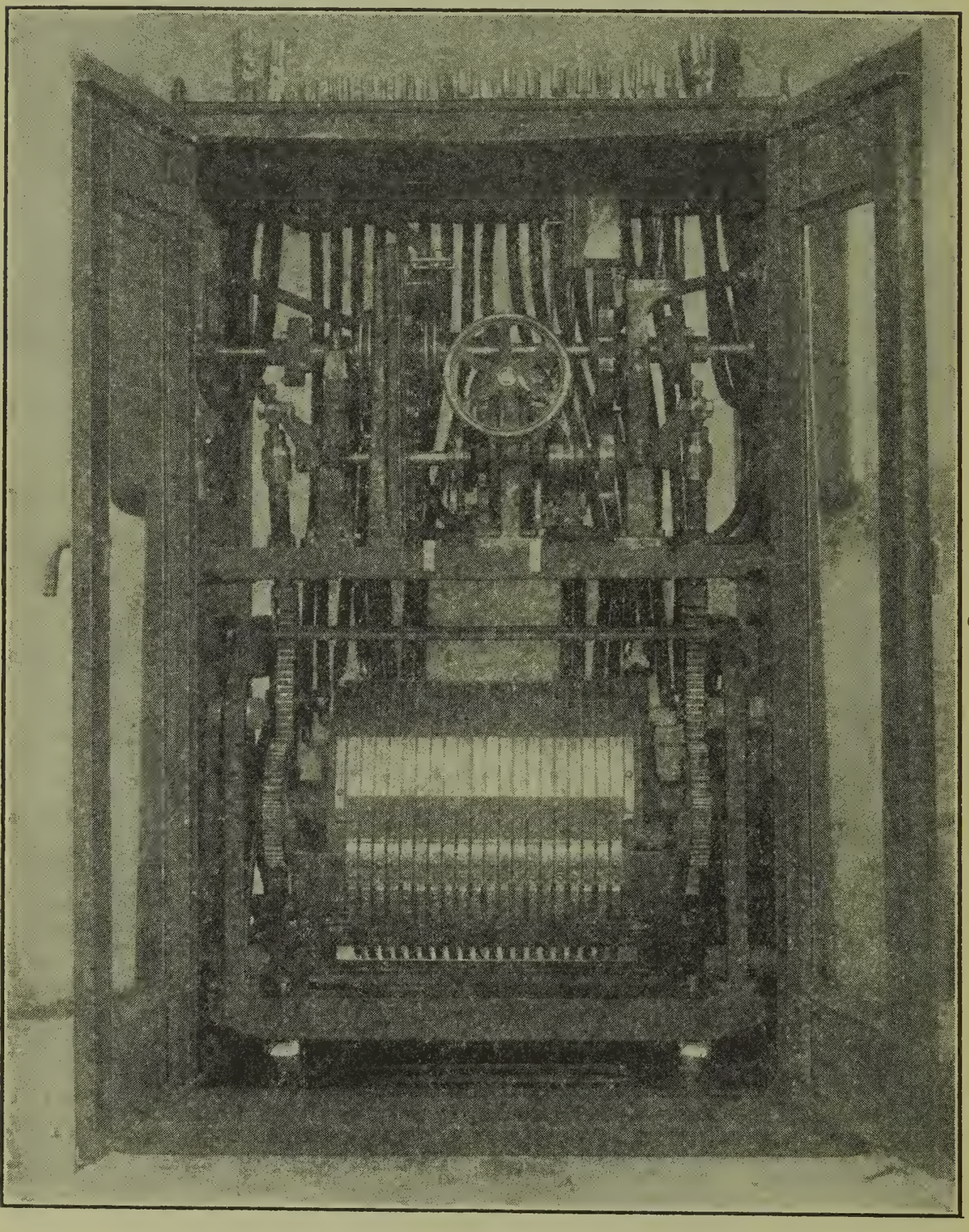

Fig. 6.

In addition to the receiving panel there are five unit panels and one direct-current control panel. The unit panels are equipped with meters and oil switches, the disconnecting switches being

mounted on a framework in the rear of the panels with the master switches controlling the units located in front.

The regulating transformers are located in dust proof brick houses at distances varying from 50 to 200 feet (17 to 67 meters) from the switch house. They are known as the unit switch type of potential regulator, the electrically operated switches being of the open type, mounted on marble panels. The control voltage is 110 volt direct current and is supplied by a small motor-generator set in the switch house.

Across the street from the carbon plant is located building No. 10, containing transformer station 10A. A small brick structure located in the center of the building houses the switchboards and transformers. The transformers are located in the basement and consist of two 1,000-kw. oil-insulated water-cooled General Electric transformers. One transformer is used for experimental purposes and has a range of 30 to 77 volts. The other transformer is used in conjunction with an induction regulator and is capable of giving a voltage variation of 45 to 60 volts.

The switchboard panels are located on the main floor, and consist of an incoming feeder panel and four unit panels, one for each transformer and regulator and one for motor transformers. The receiving panel is located along the east wall of the building and is equipped with knife-switches and General Electric oil circuit-breakers. The other panels are equipped with General Electric oil circuit-breakers, ammeters, and wattmeters.

A 2-phase, underground feeder system tapped to the same feeder that supplies station 6A enters the basement and connects to the receiving panel.

The Carborundum Company.

The main transformer station of the Carborundum Company is located in a rectangular brick building situated in the west end of the furnace room. In this building are located switchboards, water switches, two small motor-generator sets and two regulating transformers. Power is supplied to this station by ten single-conductor, lead sheathed, underground cables, each having a cross section of 1,250,000 cir. mils. (632 sq. mm.) which connect to the No. 2 generating station of The Niagara Falls Power Company.

In the east end of the station facing each other are located the two regulating transformers with their control panels. Along the south wall about four feet apart are four switchboard panels which control transformers located in bays in the furnace room and in the northwest corner of the station is a 2-phase motor panel.

The switchboard panels are standard single-phase panels, 24 inches wide, constructed of slate, made by the Westinghouse Electric & Manufacturing Company. Each panel is equipped with an automatic, 2-pole, type B circuit-breaker, knife switches and meters. Above each panel on the wall are water switches consisting of an iron tank lined with a section of sewer tile 24 inches in diameter.

The regulating transformers are four in number and bear the name of the Westinghouse Electric & Manufacturing Company. They are of the oil-insulated, water-cooled type, each with a capacity of 1,600 kw., 80 to 300 volt secondary. Two of these regulators, as mentioned before, are located in the transformer station, the other two being located in transformer houses in the furnace room.

The regulators are what is known as a unit-switch type of potential regulator, being of special design for handling heavy currents. The regulator consists of a number of electrically operated switches controlled from a master switch located in front of the control panel. The transformer windings are divided into sections and two floating coils are provided which are connected to various taps on the main auto-transformer. These floating coils have intermediate steps and the successive operation of the switches connect the floating coils in proper sequence to the main auto-transformer and transfers the line connection from one point of the floating coil to the next. The master switches are arranged with an automatic lock to prevent their being opened too rapidly. The magnet switches themselves are so interlocked that the proper sequence of operation is insured. The main contacts are oil immersed and entirely enclosed, making them dust proof.

Low-voltage control cables in underground ducts connect the master switch to the regulator, the control current being supplied from the two small motor-generator sets located in the transformer house.

In addition to the regulating transformers the Carborundum Company operates two 1,500, kw. oil-insulated, water-cooled transformers with 2,200 volt primary and 100 volt secondary, manufactured by the Westinghouse Company. These units are connected to panels in the transformer room. There is no receiving panel in the station, each single-phase circuit connecting directly to a single-phase transformer panel.

Motor power is obtained from a bank of transformers located in a shed near the furnace room, consisting of four 150 kw. General Electric oil-insulated, water-cooled transformers with 440 volt secondaries.

The No. 1 plant of the Carborundum Company is supplied from a small step-down transformer station in the basement of the mould building, consisting of two 150 kw. and six 50 kw. transformers, oil-insulated and self-cooled, made by the General Electric Company and one 75 kw., oil-insulated, self-cooled Wagner transformer. The 75 kw. transformer is used for lighting, and has a ratio of 2,200 to 110,220 volts. The other transformers supply power to motors and have 440 volt secondaries. A 2-phase standard Westinghouse panel controls the incoming power, which is supplied from No. 1 generating station of The Niagara Falls Power Company by two duplex No. 2/0 paper and lead cables.

Niagara Lead Company.

The transformer station of the Niagara Lead Company, situated in the south end of the main building, is supplied with power by The Niagara Falls Power Company by a 2-phase, 2,200-volt underground feeder system, consisting of two 3/0 duplex paper and lead cables.

The incoming cables connect to a receiving panel mounted with four disconnecting switches and two 2-pole, automatic Westinghouse type B circuit-breakers. From the receiving panel the power is carried to the motors and transformers by conductors mounted on the walls on porcelain insulators.

Direct current power is obtained from two Westinghouse motor-generator sets, each consisting of a 300 h. p., 2-phase, 2,200 volt, 487 r. p. m., induction motor, direct-connected to a 187.5 kw., 125-volt, compound-wound, direct-current generator. In addition to the motor generator sets the station contains a 60

h. p., 2-phase, 2,200-volt Westinghouse induction motor belted to an overhead line shaft, which supplies the main shop with mechanical power. This motor is protected by a panel mounted with General Electric expulsion-type fuses.

On the west wall of the station are mounted two 15-kw., oil-insulated, self-cooled, and four 5-kw., air-cooled, Westinghouse transformers, with 110-volt secondaries used for lighting. These transformers are also protected by General Electric expulsion fuses.

Each motor-generator set is provided with a motor and generator panel. The panel controlling the motor is located on the east side of the station between the wall and induction motor; ammeters, an oil-switch, auto-starter, and 4-pole, air-break circuit-breakers are mounted on the panel.

The direct-current panels are located on the opposite side of the room near the west wall. Each panel is equipped with meters, field-rheostat handle, knife-switches and circuit-breakers. The direct-current power is carried to the cell-room by underground cables.

Castner Electrolytic Alkali Company.

Power in the form of 2-phase, 2,200-volt, 25-cycle current is delivered to the Castner Electrolytic Alkali Company at three stations located in their Niagara Falls plants.

Transformer Station No. 1 has a capacity of 1,375 kw. made up of eleven 125 kw. Westinghouse rotary converters, 500 r. p. m., 2-phase, 25-cycle, with Wagner step-down, air-blast transformers, each with a capacity of 100 kw. These transformers have a secondary regulation of 30 volts, accomplished by means of a dial-switch regulator in the primary circuit. The transformers are single-phase and are connected two in a bank to each rotary. The station is rectangular in shape with the rotary converters occupying the south side and the transformers the north side. The switchboard arrangements consist of single panels mounted opposite each unit between the transformers and rotary converters. The panels are made of white marble equipped with plug-type switches and fuse-blocks. A dial-switch with glass cover is mounted on the lower part of the panel.

In the east end of the station is located a receiving panel to

which is connected eight single-conductor, lead-sheathed cables. The receiving panel is equipped with knife-switches and carbon-break circuit-breakers. There are no low-tension switchboards in the station, the direct current power being carried directly from the rotary converters to the cell room. In addition to the rotary converters the station contains four 25-kw., oil-insulated, self-cooled, Westinghouse transformers with 220-volt secondaries, which supply power to motors.

Transformer Station No. 2 has a capacity of 4,000 kw., consisting of eight 500-kw. Stanley synchronous-motor, direct-current generator sets, the motors being 2-phase, 2,200-volts and rated at 580 kw. The sets are arranged four in a row on each side of the transformer room. On opposite sides of the room, between the wall and motor-generator sets, are two eight-panel marble switchboards made by the Stanley Manufacturing Company. Each switchboard consists of four alternating-current panels and four direct-current panels, mounted side by side. The alternating-current panels are equipped with oil circuit-breakers, Stanley hot-wire ammeters and field ammeters. The direct-current panel is equipped with Weston ammeter, volt-meter, knife-switches, and circuit-breakers. Supported on brackets in the rear of the switchboard are both alternating and direct-current bus-bars. Located in the northwest corner of the station is the main-line panel, equipped with eight knife-switches and carbon-break circuit-breakers. The incoming cables, eight in number, have a cross section of 1,000,000 cir, mils. (506 sq. mm.) each and are capable of transmitting approximately 8,000 h. p.

Motor power for the No. 2 station is obtained from six 50-kw. Wagner transformers with 2,200-volt primary and 220-volt secondary. A 75-kw., oil-insulated, self-cooled Wagner transformer supplies power for lighting. These transformers are controlled from a panel equipped with oil circuit-breakers.

No. 3 station of the Castner Company is equipped with an 800-kw. General Electric, oil-insulated, water-cooled, transformer with 2,200-volt primary and 144-volt secondary. Connected in the secondary circuit is a potential regulator capable of varying the voltage from 85 to 225 volts. The regulator is oil-insulated, water-cooled, and of same make as the transformer. A single-phase slate switchboard is mounted in the station in front of the

transformer. The switchboard is equipped with knife-switches, oil circuit-breakers, indicating meters and watt-hour meters. The primary circuit is opened and closed through a water-switch, mounted on the wall above the transformer.

The Norton Company.

The electrical equipment of the Norton Company at its Niagara Falls, N. Y., works is typical of the equipment of several plants in the Niagara district. Power is received at a switch house, from which it is distributed to step-down transformers located in bays along the side of the furnace room.

In the switch house are located a marble receiving panel mounted with disconnecting switches, two double-pole, General Electric, K3, oil circuit-breakers, a motor and light panel equipped with knife-switches and General Electric expulsion fuses, and a transformer panel. Adjacent to the switch house is a transformer bay containing a 1,500-kw., oil-insulated, water-cooled, Wagner transformer with 2,200-4,400 volt primary and 110-220 volt secondary, with a water-switch mounted on the wall above the transformer. The panel for this transformer is located in the switch house.

Attached to the furnace room on the same side as the switch house are two bays containing 750-kw. units, together with control-panels and water-switches. One of these transformers bears a Westinghouse name plate, the other a General Electric. Both units have a ratio of 2,200 to 110 volts, and are of the oil-insulated, water-cooled, type. The bays are connected to the switch-house by a pair of single-conductor, lead-sheathed cables with taps at each bay.

In the north end of the main building are located the lighting and motor transformers, consisting of two 30-kw. and two 10-kw., oil-insulated, self-cooled, Westinghouse transformers with 110 and 440-volt secondaries.

Power is supplied to the Norton Company from No. 2 generating station of The Niagara Falls Power Company by a two-phase, 2,200-volt feeder system, consisting of two 500,000 cir. mil. (253 sq. mm.) and two 1,000,000 cir mil. (506 sq. mm.) lead-sheathed cables. The secondary current of the three furnace units is carried to the furnace room by copper bus-bars inter-

laced up to the furnaces. Each furnace is equipped with an indicating ammeter mounted on the wall opposite the furnace.

Union Carbide Company.

The plant of the Union Carbide Company at Niagara Falls, N. Y., is of interest both on account of its size and on account of the great amount of power transformed by it for electrochemical purposes.

Power is distributed to the Union Carbide Company by The Niagara Falls Power Company and by the Cliff Electrical Distributing Company, from sub-stations located near the Carbide Works.

The Union Street sub-station of The Niagara Falls Power Company is located on the west side of the Carbide Company's No. 1 plant at a distance of about 60 feet. This station is used as a switching station and as a step-down station, being equipped with switch-board panels, oil circuit-breakers, and step-down transformers (Fig. 7).

The sub-station of the Cliff Electrical Distributing Company is located midway between the No. 1 and No. 2 plants and contains switching apparatus only.

In the plants of the Carbide Company, widely separated from the sub-stations, are the large low-voltage step-down transformers which supply power to the furnaces. Some of the transformers have 2,200-volt primaries and others 11,000-volt primaries. They are all of the oil-insulated, water-cooled type, the cooling water being procured from the city mains.

Practically all of the 11,000-volt units have a double source of supply for their power. Bricked in the wall of the transformer bay is a marble slab mounted with a double-throw knife-switch, the middle point of which is connected to the transformer, the jaws of one side being connected to The Niagara Falls Power Company's circuits and those on the other side to the Cliff Electrical Distributing Company's circuits.

The Union Street sub-station of The Niagara Falls Power Company has a capacity of 52,000 h. p., made up of four 1,870-kw. step-down General Electric transformers, two 11,000-volt switchboards, and one 2,000-volt switchboard, and supplies power to all of the plants in the immediate vicinity, including that of

Union Carbide Company. Power is delivered to the station by eleven 3/0, 3-conductor, paper and lead cables, and a 3-phase, 500,000 cir. mil. (253 sq. mm.) aluminum overhead transmission line, both of which feed into a double bus-bar system through panels mounted with double-throw selector switches.

The switching apparatus which controls the carbide units is of two kinds: 2,000-volt and 11,000-volt apparatus. The 11,000-volt units are fed from panels equipped with ammeters, switch-

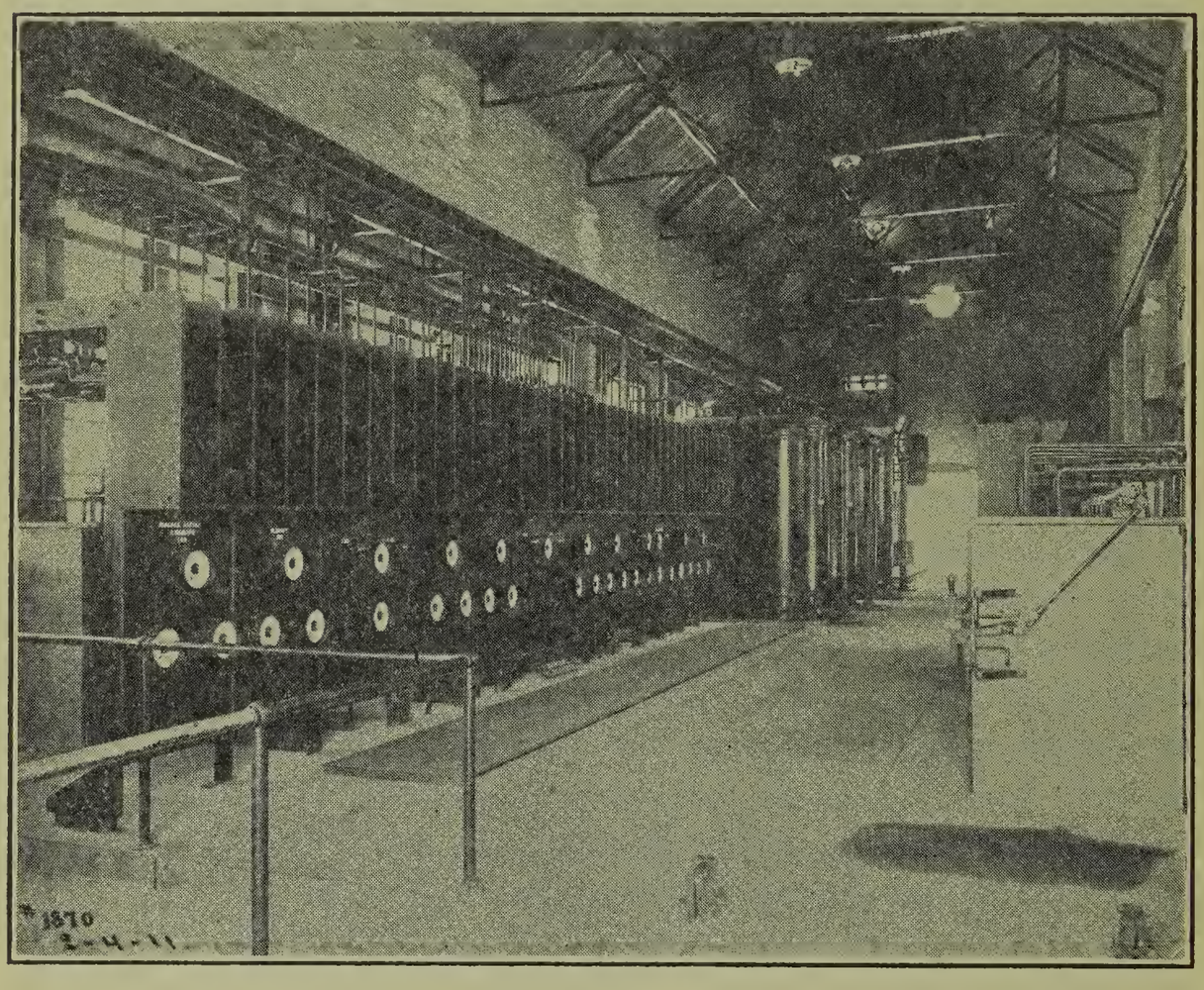

FIG. 7.

handles, and double-throw, single-pole knife-switches mounted on electrose insulators. The brick cells containing 2-pole, General Electric, K4, oil-switches are mounted back of the panels along the wall. Each unit is connected to the station by a duplex paper and lead cable laid in underground ducts.

The 2,000-volt units are fed from a switchboard on the east side of the station. Each unit is connected to a single-phase panel mounted with ammeters, oil circuit-breakers and selector-switches.

The substation of the Cliff Electrical Distributing Company consists of a one-story brick building with basement. Power enters the station at a pressure of 12,000 volts over five 3-conductor, paper and lead cables through ducts at the South end of basement (Fig. 8).

Each cable terminates in a pot-head, thence passes up to the main floor to knife-switches mounted in compartments in the rear of type C, Westinghouse-automatic, oil, circuit-breakers. From

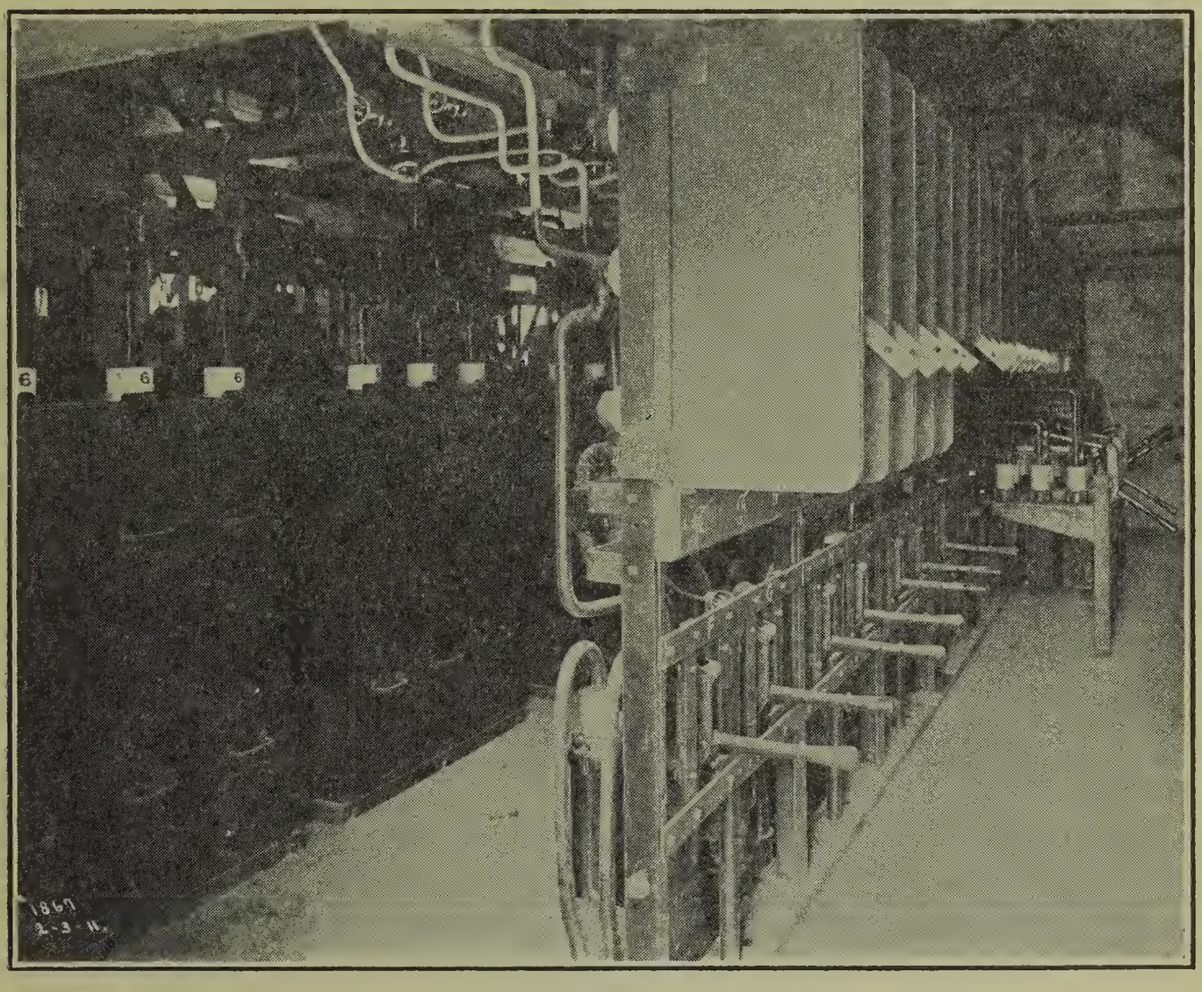

Fig. 8.

the knife-switches the circuits pass through the oil-switches to knife-switches and thence to bus-bars mounted on a maple framework.

The outgoing cables are connected to the bus-bars through knife-switches and non-automatic oil circuit-breakers.

Niagara Alkali Company.

The new transformer station (Fig. 9) of the Niagara Alkali Company will have, when completed, a capacity of approximately

1,500 kw., made up of two 750-kw., rotary converters, 6-phase, 375 r. p. m., direct-current voltage, 205 to 235 volts—one manufactured by the General Electric Company (Figs. 10 and 11) and the other by the Westinghouse Electric and Manufacturing Company (Fig. 12). The General Electric unit is known as a regulating-pole rotary converter, the direct-current voltage being varied by changing the excitation of the auxiliary poles. The Westinghouse unit regulates the direct-current voltage by a synchronous regu-

Fig. 9.

lator mounted on the same shaft as the rotary converter, and connected between the armature-windings and collector-rings. Both these rotary converters have high synchronizing power and will not drop out of step when the alternating current voltage is dropped to 40 percent of normal.

Power is received over a 3/0, 3-conductor, 11,000-volt, paper and lead-covered cable from the Union Street sub-station of The Niagara Falls Power Company, and stepped down in 3-phase,

870-kw., oil-insulated, water-cooled transformers to a voltage suitable for the rotary converters.

The high-tension switching apparatus consists of four 3-phase, automatic, K4, 15,000-volt General Electric oil circuit-breakers, mounted in cells, constructed of pressed brick ranged along the west wall of the transformer station. One switch controls the main-line, the other three are unit switches for control of the two rotary converters and a bank of motor and light transformers.

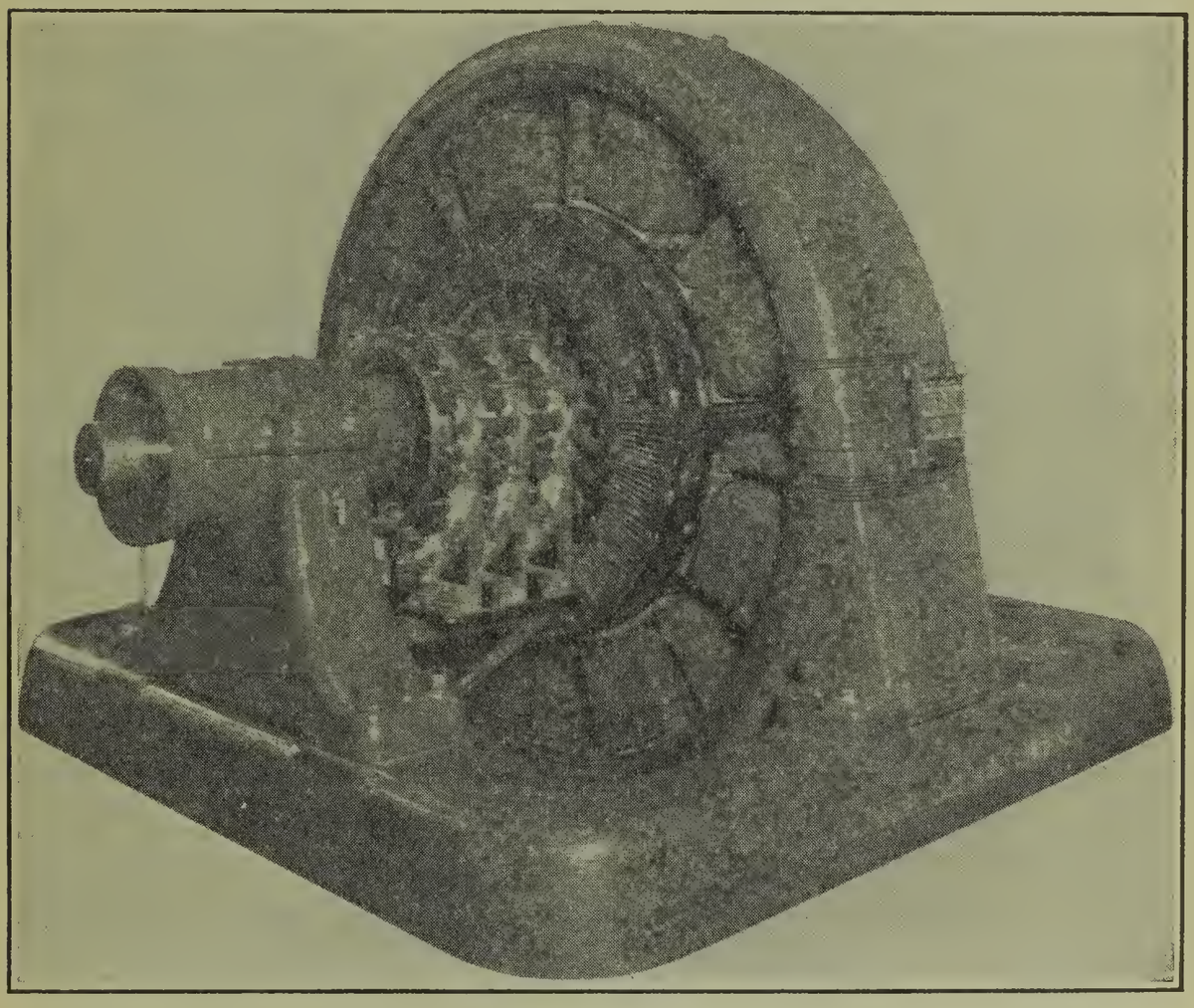

Fig. 10.

Above the oil switch cells is an iron-pipe structure carrying the bus-bars, meter-transformers and disconnecting-switches. The bus-bars consist of heavy-insulated rubber and cambric cables, mounted on petticoat insulators. Switch-handles for operating the oil-switches are mounted on the side walls of the brick cells. A main-line disconnecting panel is fastened to the wall directly above a man-hole in the north-west corner of the station. Between the rotary converters and transformers are two starting panels

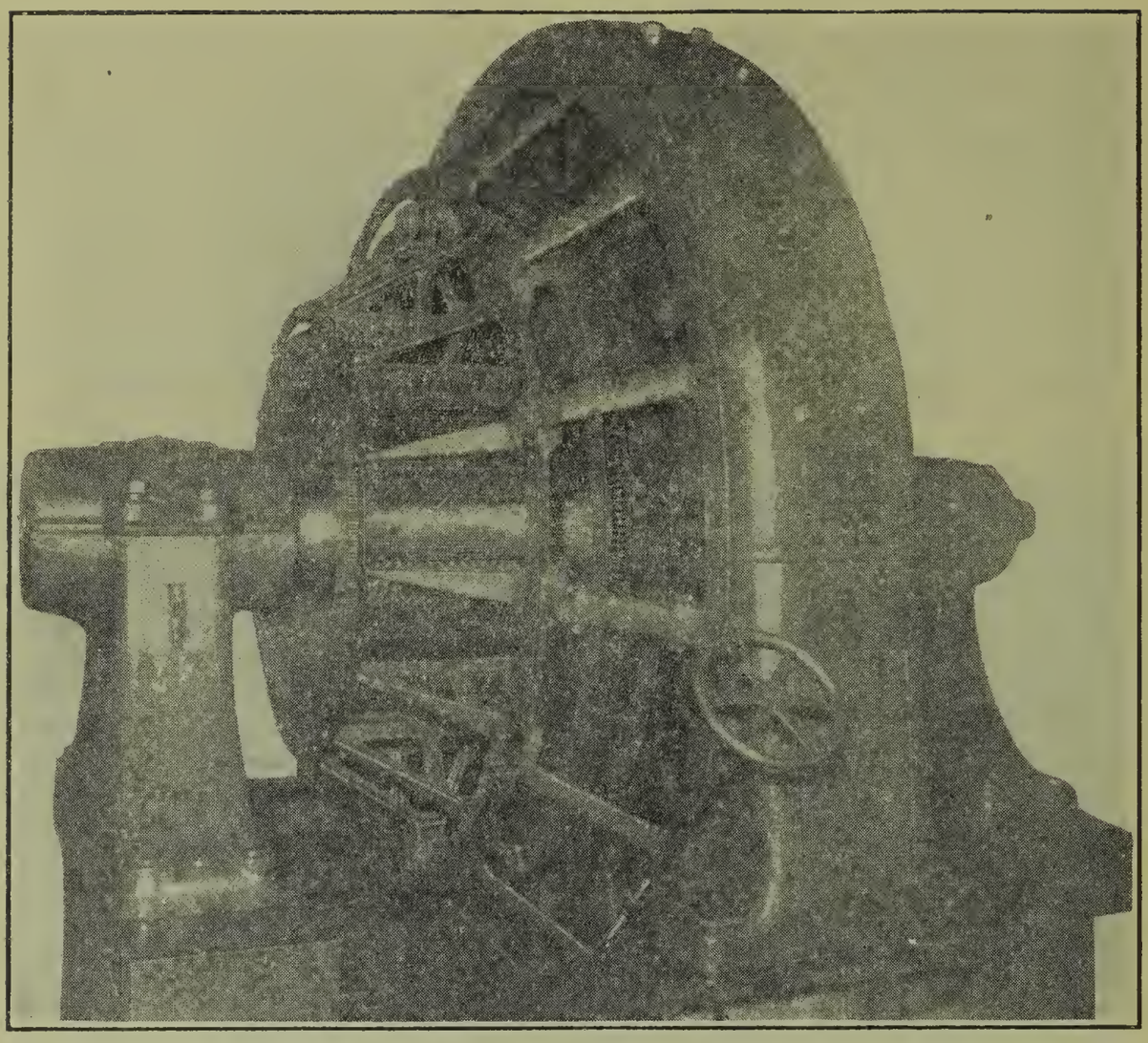

FIG. 11.

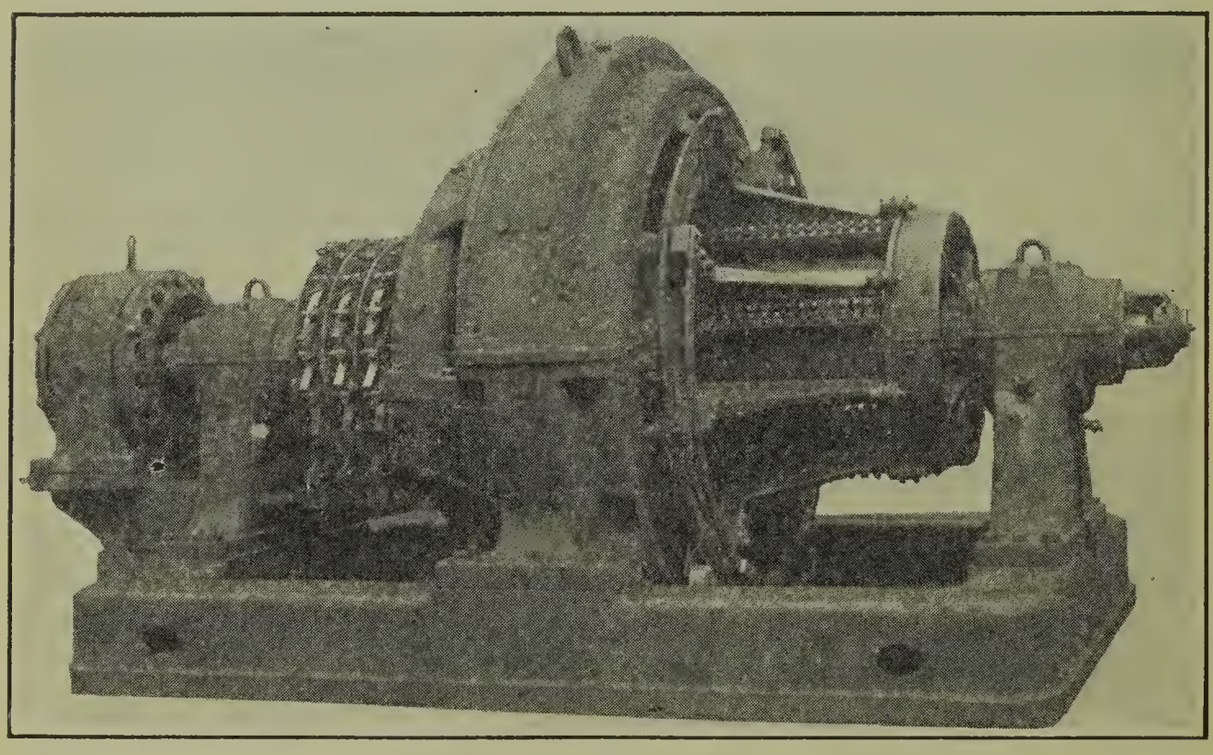

FIG. 12.

mounted with 3-pole, double-throw knife-switches. On the east side of the room is a low-tension switchboard consisting of slate panels 24 inches wide. Two panels control the direct-current side of the rotary converters, and are equipped with knife-switches, circuit-breakers, indicating-voltmeter and ammeters. The other panels contain the high-tension indicating-meters and watt-hour-meters.

In addition to the rotary converters the station will contain two 25-kw. and two 50-kw., oil-insulated, self-cooled, General Electric transformers with a ratio of 11,000 to 460 volts, for motor power, and a lighting transformer of 15 kw. capacity, oil-insulated, self-cooled, made by the General Electric Company, with a secondary voltage of 115 to 230 volts. All the low tension wiring is carried underground in fibre conduit. An overhead crane of 10 tons capacity serves the station. A pit is excavated in the northwest corner of the station, so that transformers can be readily dismantled and repaired.

Hooker Electrochemical Company.

The power station of the Hooker Electrochemical Company, recently reconstructed and enlarged, presents a good example of concrete construction as applied to switchboard work (Fig. 13). The station is located in a brick building with a basement floor. Power enters the building in the form of 3-phase, 11,000-volt, 25-cycle current, over two 3-conductor, 3/0 lead-sheath, paper-insulated cables which separate into six single-conductor cables in the basement and thence pass up through the floor to disconnecting switches mounted in concrete cells on the main floor. On the south wall of the building is located the concrete structure which contains the oil-switches and bus-bars. The cells containing the Westinghouse type E oil-switches are built on the floor. Above them are the disconnecting-switch and bus-bar compartments, the latter running lengthwise of the room. The bus-bars consist of bare wire, 4/0 in cross section, mounted on petticoat insulators. The oil switches are remote-control, hand-operated, 300-ampere capacity. The high-tension switchboard panels, six in number, are of blue Vermont marble and are equipped with power-factor meters, ammeters, and switch handles. All the meter-transformers are mounted in concrete cells, the low-tension circuits of

these transformers being carried to the switchboards in iron conduits.

The 11,000-volt current is stepped down by 300 and 400 kw. oil-insulated, water-cooled, single-phase Westinghouse transformers, located in a bay on the north side of the station. The 300-kw. transformers have a ratio of 11,000 volts to 198 volts and are connected in *delta,* supplying power to rotary converters. Some of the 300-kw. transformers are arranged for 3-phase to 2-phase

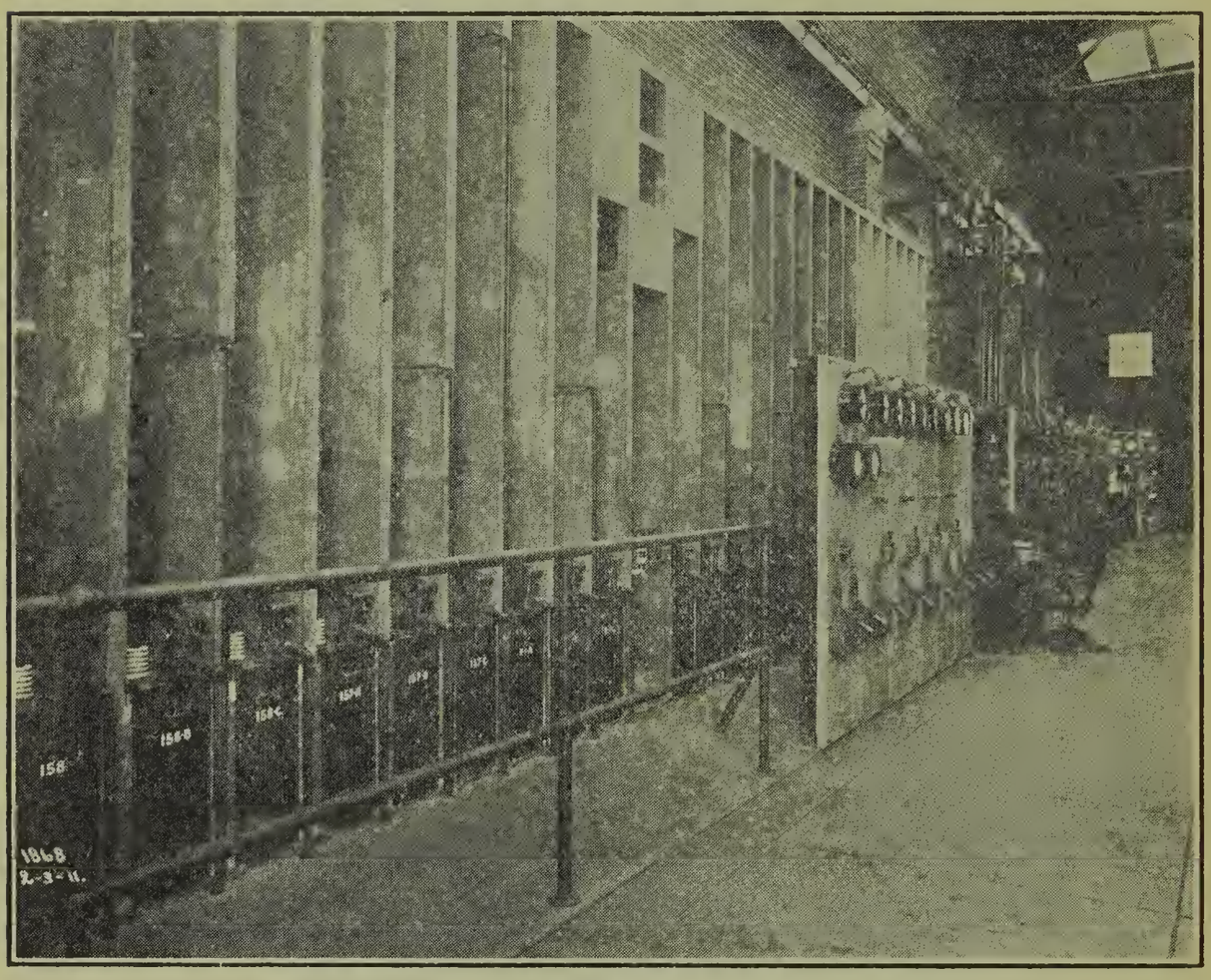

FIG. 13.

transformation and have a secondary voltage of 2,200 volts. The 400-kw. transformers have a secondary voltage of 220 volts, and are used for lighting and motor power.

Direct current power is obtained from Westinghouse booster-type rotary converters (Fig. 14), 6-phase, 375 r. p. m., direct-current voltage 230 to 320; and from Burke Electric motor-generator sets consisting of 720 h. p. synchronous motors, 2-phase, 2,200 volts, 375 r. p. m., direct connected to 500-kw., 250-volt, direct-current generators, and 360 h. p., 2-phase, 2,200-volt syn-

chronous motors, direct connected to 250-kw., 125-volt, 500 r. p. m., direct-current generators. The synchronous motors are excited by two Burke Electric exciter sets consisting of a 90 h. p. induction-motor, 2-phase, 2,200 volts, direct connected to a 60-kw., 125-volt, 720 r. p. m., direct-current generator. All the machines in the station are arranged so that they can be operated in multiple on the direct-current side, a direct-current bus-bar being carried along the rear of the low-tension switchboard. Each

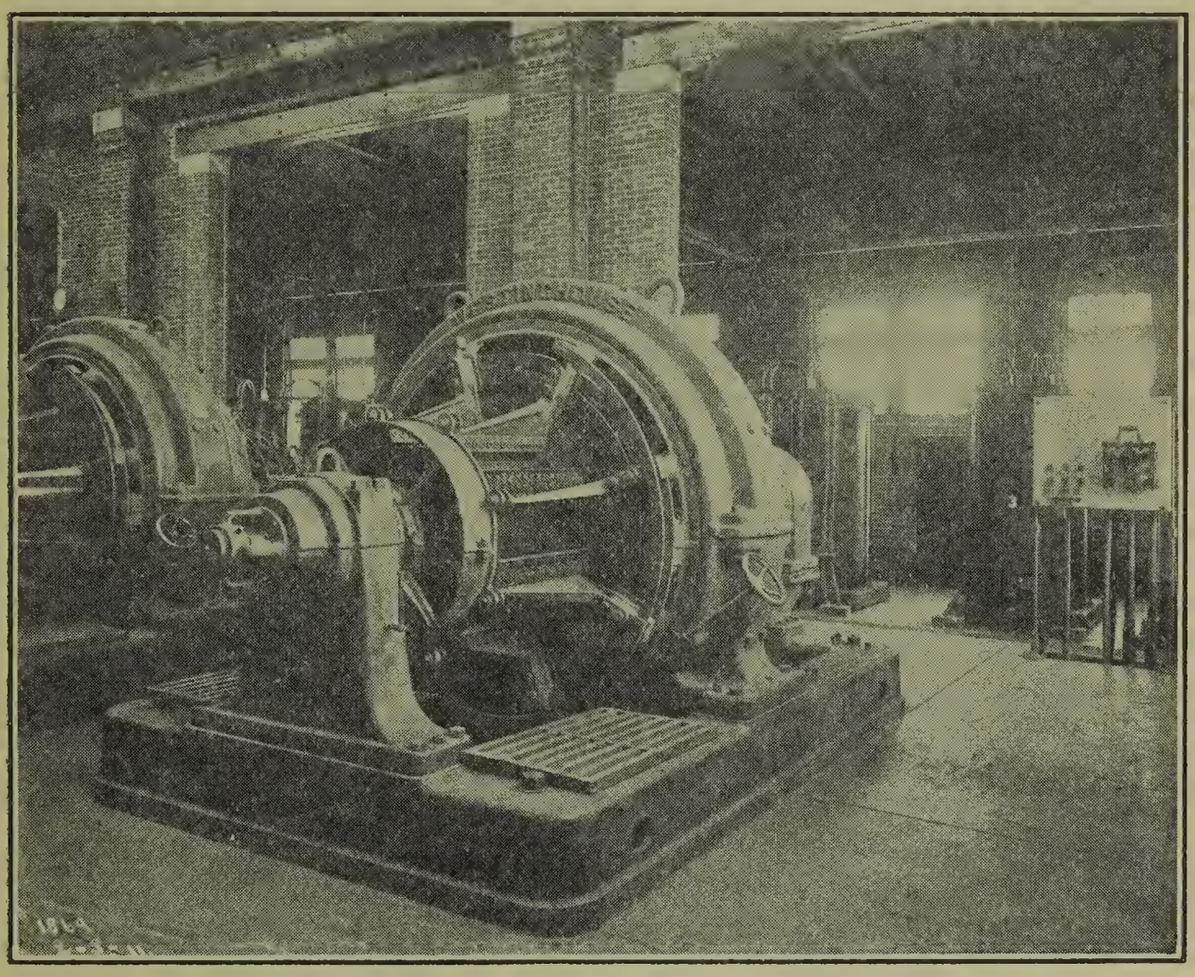

FIG. 14.

machine is controlled by a low-tension panel which is equipped with circuit-breakers, knife-switches and ammeters. The Burke Electric machines are controlled on the 2,200-volt side by panels equipped with oil-switches.

The low-tension circuits of the motor and light-transformers are carried to six panels mounted with four-pole knife-switches and cartridge fuses. A 2,200-volt receiving panel through which power was formerly supplied to the Burke machines is still in service, being used as a spare. The rotary converters are

synchronized by small induction-motors mounted on the end of the shaft. A low-tension starting panel, mounted with two 3-pole knife-switches, is connected in the circuit between the transformers and alternating-current side of the converters. All of the 11,000-volt apparatus was furnished and installed by the Westinghouse Electric and Manufacturing Company.

Niagara Electro-Chemical Company.

The transformer station of the Niagara Electro-Chemical Company is of interest in that it contains several different types of apparatus, each of which performs the same service, but in a slightly different manner.

The function of the station is to change the 2-phase, 2,200-volt, 25-cycle current received from The Niagara Falls Power Company into direct-current power. To perform this transformation the station is equipped with three different types of apparatus, namely, rotary converters with regulating transformers, induction motor-generator sets, and synchronous motor-generator sets.

The transformer station is a large roomy brick building situated on the east side of the cell room. The general scheme carried out in the station is that of locating the transformer units on the center line of the building with the switchboards along the walls. The receiving panels being located along the east wall and the unit panels along the west wall.

Power is delivered to the station by two 2-phase, underground feeder systems, one of which connects to a receiving panel in the south end of the station and the other to a panel in the north end of the station. A third feeder enters the building and connects to both panels. This feeder is considered a spare feeder and is used only in times of trouble on one of the other feeders. The panel in the south end receives its power from No. 1 generating station over four cables with a cross section of 1,000,000 cir. mils. (506 sq. mm.) each. The north end panel is supplied from No. 2 generating station, the cables having a cross section of 1,250,000 cir. mil. (633 sq. mm.).

Both of the receiving panels are of marble, and are equipped with eight knife-switches, single-throw, single-pole, and two double-pole Westinghouse type B, automatic circuit-breakers.

From the receiving panels the power is carried to the unit panels through underground ducts in the floor of the station. The rotary converters in the south end of the station are equipped with starting motors and polyphase Stillwell regulators in the primary of the transformer circuits. The static transformers are of the oil-insulated, self-cooled type, both rotary converters and transformers being made by the Westinghouse Company. Each rotary is connected to a marble panel on both the low tension and high tension side. The induction-motor-generator sets are of standard design made by the Westinghouse Company. Each set is connected to two marble panels mounted with the usual instruments and switches.

The synchronous-motor-generator sets are made up of three units, two direct-current generators connected to opposite ends of a synchronous-motor. Each machine is connected to a separate panel mounted with meters and circuit-breakers. The switchboard is of blue Vermont marble, designed and installed by the Westinghouse Electric and Manufacturing Company.

The United States Light and Heating Company.

Located in the north end of the City of Niagara Falls is The United States Light and Heating Company, which has just completed a new transformer station designed by Dodge, Day & Zimmerman of Philadelphia. When completed the station will contain four 750-kw. rotary converters, with necessary step-down transformers; two sets have already been installed (Fig. 15).

At the present time power is received from the Cliff Electrical Distributing Company by an over-head transmission line which will soon be replaced by an underground cable system. Power enters the station in the form of 3-phase, 25-cycle current, at a pressure of 12,000 volts, and passes through General Electric disconnecting-switches mounted on insulators on a pipe framework adjoining the south wall of the station. On each side of this iron structure are mounted meter-transformers and fuses. From the disconnecting switches circuits are carried overhead to choke-coils located on a pipe framework mounted on top of a 3-pole, cell-type, K12, General Electric, 15,000-volt, oil circuit-breaker. From this support the circuits pass through the oil-switch to a bus-bar system mounted directly over the static transformers supported on insulators on an iron framework.

In front of the K12, oil circuit-breaker stands a blue Vermont-marble panel equipped with Thompson horizontal-type ammeter, watt-meter, power-factor meter, time-limit relay, and switch-handle. General Electric meter-transformers are mounted on the framework above the oil-switch cells.

In the rear and at right angles to the main oil-switch structure, facing in opposite directions are two additional brick structures containing automatic, remote-control, 3-pole, 15,000-volt,

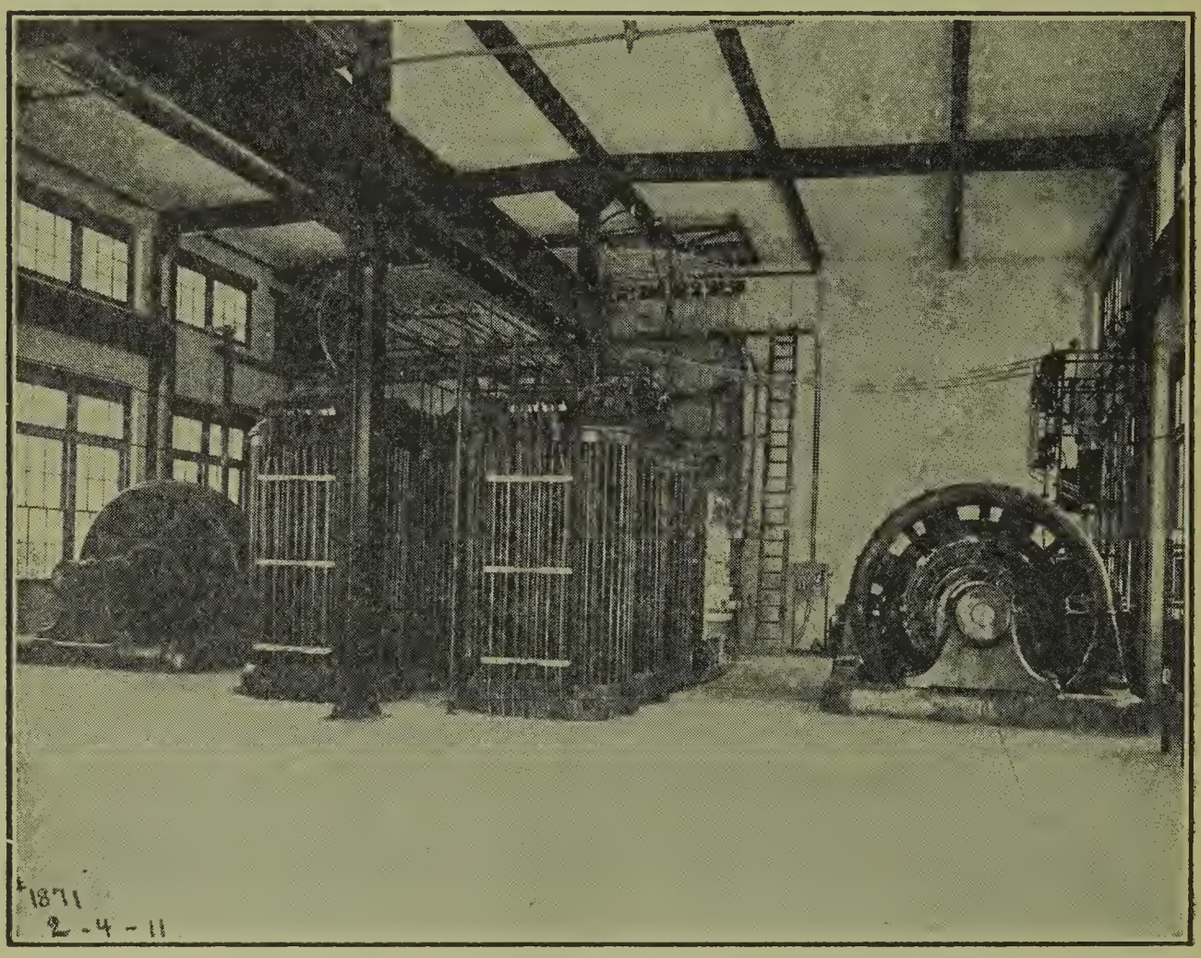

FIG. 15.

K4, General Electric oil-switches, which control the primary circuits of the static transformers. Above each cell is a pipe frame work on which is mounted meter-transformers.

From the main-line oil-switch the current divides and passes through each of the two oil-switches described above to the primaries of the two banks of transformers. Located in front of the switch-cells are three marble panels, one of which controls the oil-switch, another being a starting panel for a rotary converter, while the third has mounted upon it the neutral circuit-breakers.

On the oil-switch panel are mounted Thompson volt-meter, ammeter, power-factor-indicator, time-limit relay and switch handle. On the starting panel are mounted a 2,500-ampere, 3-pole, double-throw knife-switch, and a 1,200-ampere, 3-pole, double-throw knife-switch. On the rear of this panel is mounted a 3-pole, carbon-break circuit-breaker in the middle point of the secondary winding of the transformers.

Two banks of transformers, each bank consisting of three 275-

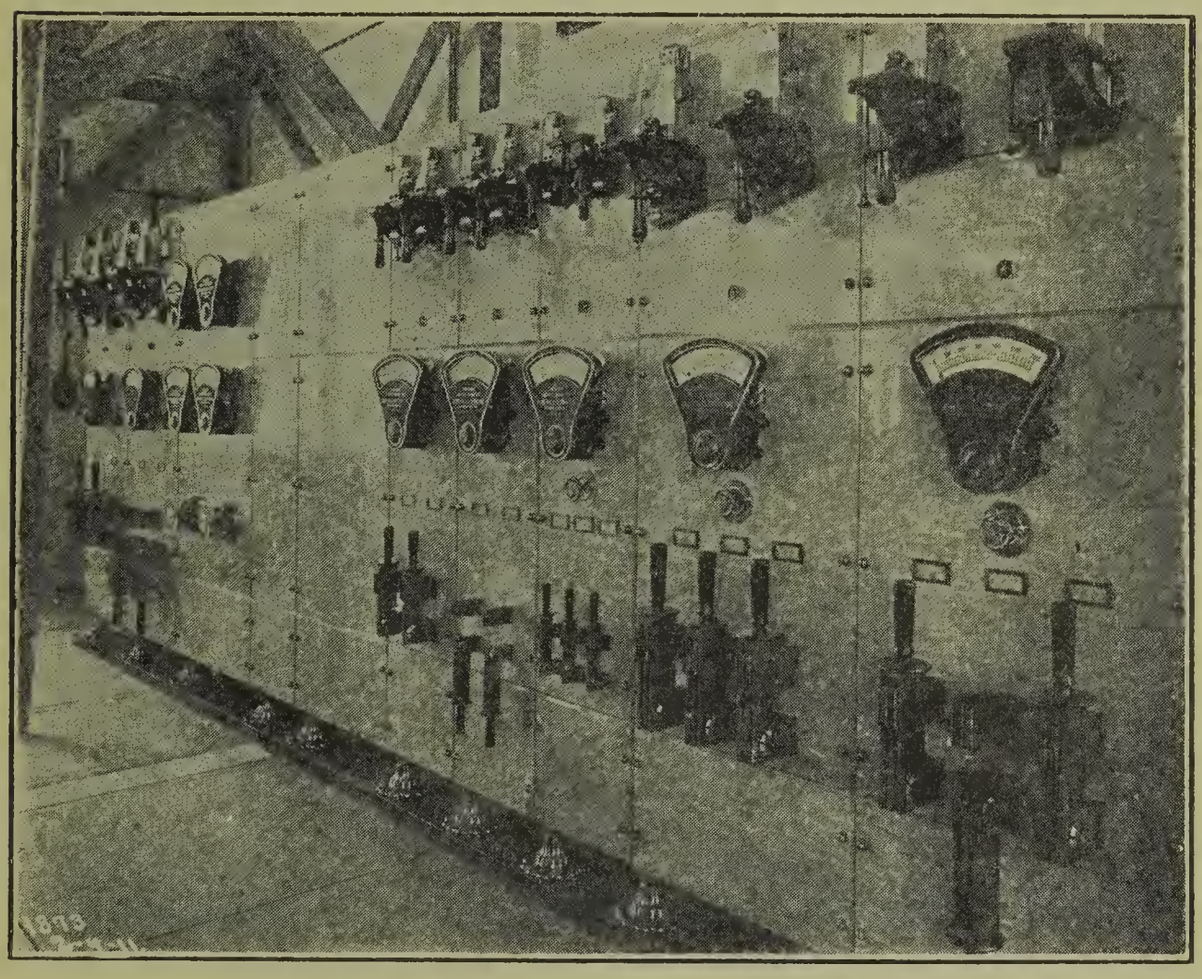

Fig. 16.

kw., single-phase, oil-insulated, self-cooled, General Electric transformers, with 6,930-volt primaries, Y-connected, and 199-volt secondaries, step-down the current for use in the rotary converters. The secondary leads of the transformers are carried to the starting panels, thence to the rotary converters through underground trenches on single-conductor, lead-covered cables.

The 750-kw., 6-phase, split-pole, 375 r. p. m., General Electric rotary converters with a direct-current voltage of 225 to 275 volts occupy space on each side of the station. The main and

auxiliary field rheostats are mounted on angle iron framework near the rotary converters. Direct current power from the rotary converters is carried to a switchboard in the east end of the station through underground ducts by single-conductor lead-covered cables.

The direct-current switchboard (Fig. 16) consists of ten blue Vermont marble panels 2½ in. (6.2 cm.) thick, made by the Fort Wayne Electric Works. On the left hand end, facing the switchboard, are two 30-in. (75 cm.) panels which control the direct-current side of the two rotary converters, each panel being equipped with two carbon-break circuit-breakers, two single-pole, single-throw, 5,000-ampere knife-switches, and a 6,000-ampere Thompson astatic ammeter. Next to the rotary panel is a 36-in. (90 cm.) panel equipped with three 300-volt Thompson astatic voltmeters, one 10,000-ampere ammeter and two Sangamo watt-hour meters. Two vacant panels come next, one 18 in. (45 cm.) wide and the other 30 in. (75 cm.) wide, which have been reserved for booster controlling switch and instruments. A motor panel occupies the space beside the vacant 30-in. (75 cm.) panel. This panel is 18 in. (45 cm.) wide, and is mounted with two knife-switches, 1,500-ampere ammeters and two carbon-break circuit-breakers. An 18-in. (45 cm.) fire-pump panel joins the motor panel, equipped with an 800-ampere ammeter, two single-pole, single-throw knife-switches and two carbon-break circuit-breakers. The eighth panel is an 18-in. (45 cm.) lighting panel, equipped with a 600-ampere ammeter, 2-pole circuit-breaker and a 3-pole, single-throw, 400-ampere knife-switch. The ninth and tenth panels are what is known as battery panels. These are each equipped with one 3,000-ampere ammeter, one 2-pole circuit-breaker and three 3,000-ampere, single-pole, single-throw knife-switches.

In the rear of the switchboard are located the copper bus bars, three in number, constructed of 10 by ¼ in. (25 x 0.62 cm.) copper bars. Three bus-bars are required, as the plant is operated on a three-wire system. From this switchboard the power is carried to the shops by insulated copper cables supported on an overhead pole line. The station is served by two overhead cranes of sufficient capacity to handle any unit in the station.

Electro Metals Company.

At Welland, Ontario, the Electric Metals Company has installed a modern transformer station which steps-down the high voltage current received from Niagara Falls to a low voltage suitable for furnace operation.

Power is received from Niagara Falls over two-pole lines, each carrying two 3-conductor transmission lines which are dead-ended at a switch platform located in the street outside of the Electro

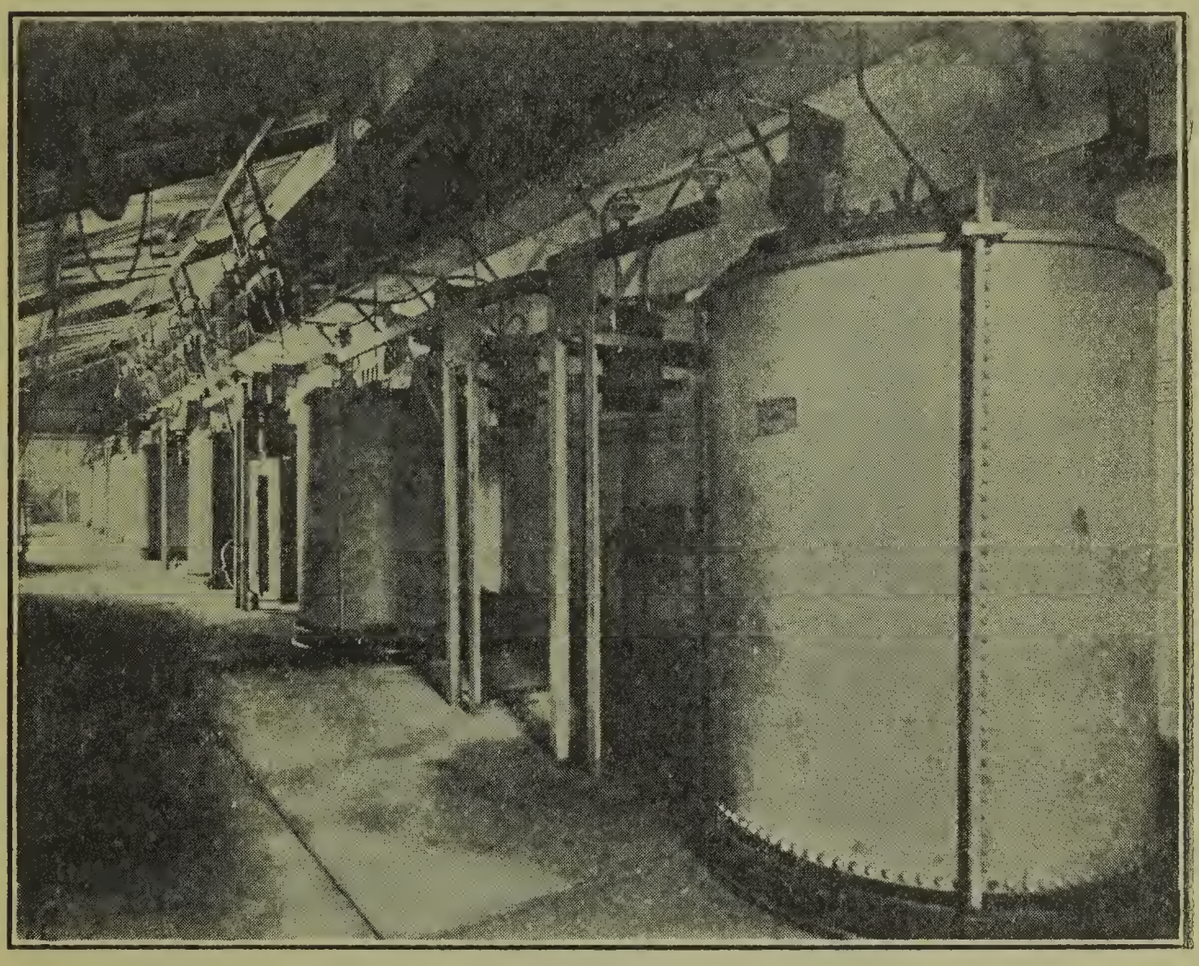

Fig. 17.

Metals Company's plant. From this structure the current is conducted into the plant over a single-pole line carrying two 3-conductor circuits which enter a brick switch-room through porcelain tubes fastened in the wall and connect to knife-switches mounted directly inside on the wall. From the knife-switches the circuits are carried to bus-bars, thence through choke-coils to a 3-pole, type H, 13,000-volt Canadian General Electric Company oil circuit-breaker, mounted in a concrete cell on the main floor.

Mounted on the east wall is a meter panel containing three

Westinghouse type F ammeters, watt-hour meters, and a Westinghouse graphic wattmeter. On the floor to the left of the meter board is a concrete structure containing the meter potential transformers and fuses. Westinghouse electrolytic lightning arresters protect the incoming lines.

From the bottom of the oil-switch the circuits pass along the west wall of the switch-house to the main transformer room, where they connect to bus-bars supported on three concrete shelves built in the wall above the transformers. On a shelf on the west wall of the switch-house are three 25-kw. Packard oil-insulated, self-cooled transformers with a ratio of 12,000 to 220 volts, which supply power to the motor circuits. These transformers are protected by a General Electric 3-pole, K4, oil circuit-breaker (Fig. 17).

In the main transformer room are located seven 750-kw. units with 12,000-volt primaries and 40-volt secondaries, oil-insulated, water-cooled, manufactured by the Packard Company. They are arranged in a line along the wall which separates the transformer room from the furnace room. Each transformer is controlled by a 2-pole, K4, automatic, remote-control, oil circuit-breaker in one leg of the circuit, the other leg being connected to the bus-bar through a knife-switch. The circuit-breaker is mounted on a wooden framework alongside the transformer, and is controlled from the furnace room by iron connecting-rods. Knife-switches are mounted above the transformers, arranged to connect the transformers to any one of the three bus-bars, thereby keeping the load balanced. Copper bus-bars connect the transformers to the furnaces, the load on the furnace being regulated by a Thury regulator, manufactured in Geneva, Switzerland. A 500-kw. Packard water-cooled transformer is used for experimental purposes.

Norton Company.

The Norton Company has recently completed a new plant at Chippawa, Ontario, for the manufacture of crystolon. In this plant a modern transformer station has been installed whose function is to transform the high-voltage power generated by the Ontario Power Company at Niagara Falls to a lower voltage suitable for furnace work and induction-motor operation.

The transformer station proper is divided into two sections, namely, a room for the transformers and a room for the main-line switches, lightning-arresters, etc. The 3-phase transmission lines carried on the same pole line terminate in the switch-house and connect to knife-switches, cross-connected on the lower side so as to parallel the lines whenever both sets of switches are

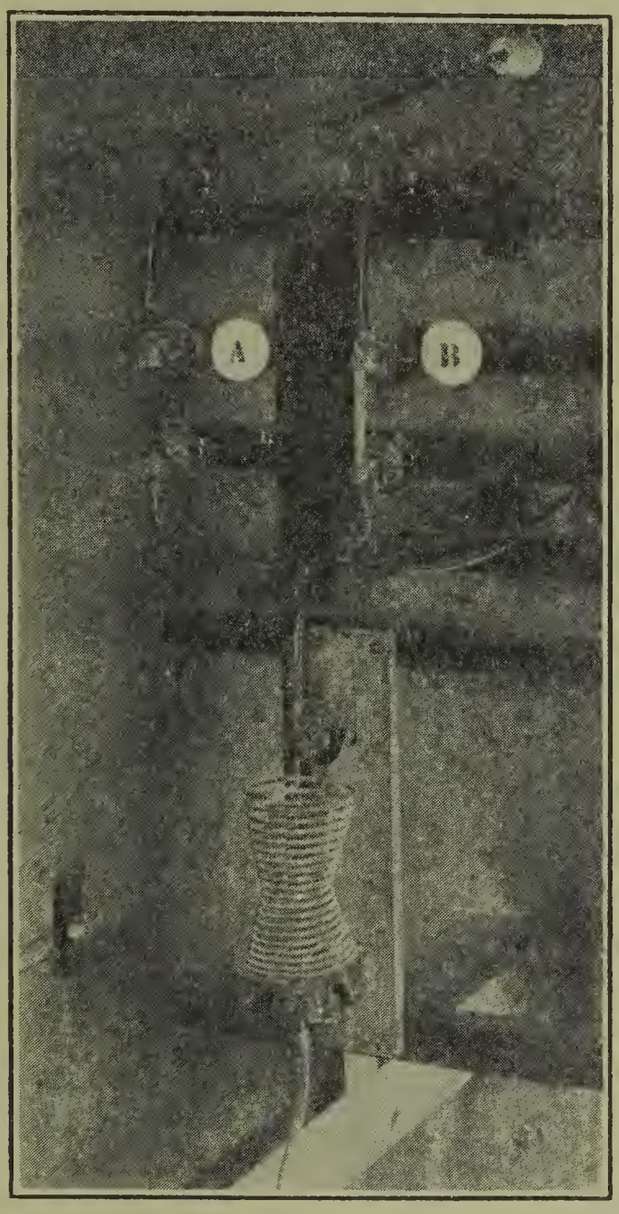

Fig. 18.

closed. From the knife-switches the current is conducted through choke-coils (Fig. 18) to a 3-pole, 300-ampere, form H4, 13,000-volt Canadian General Electric Company oil circuit-breaker (Fig. 19) mounted in a concrete cell on a gallery on opposite side of switch-house. This gallery is reached from the main floor by a spiral stairway.

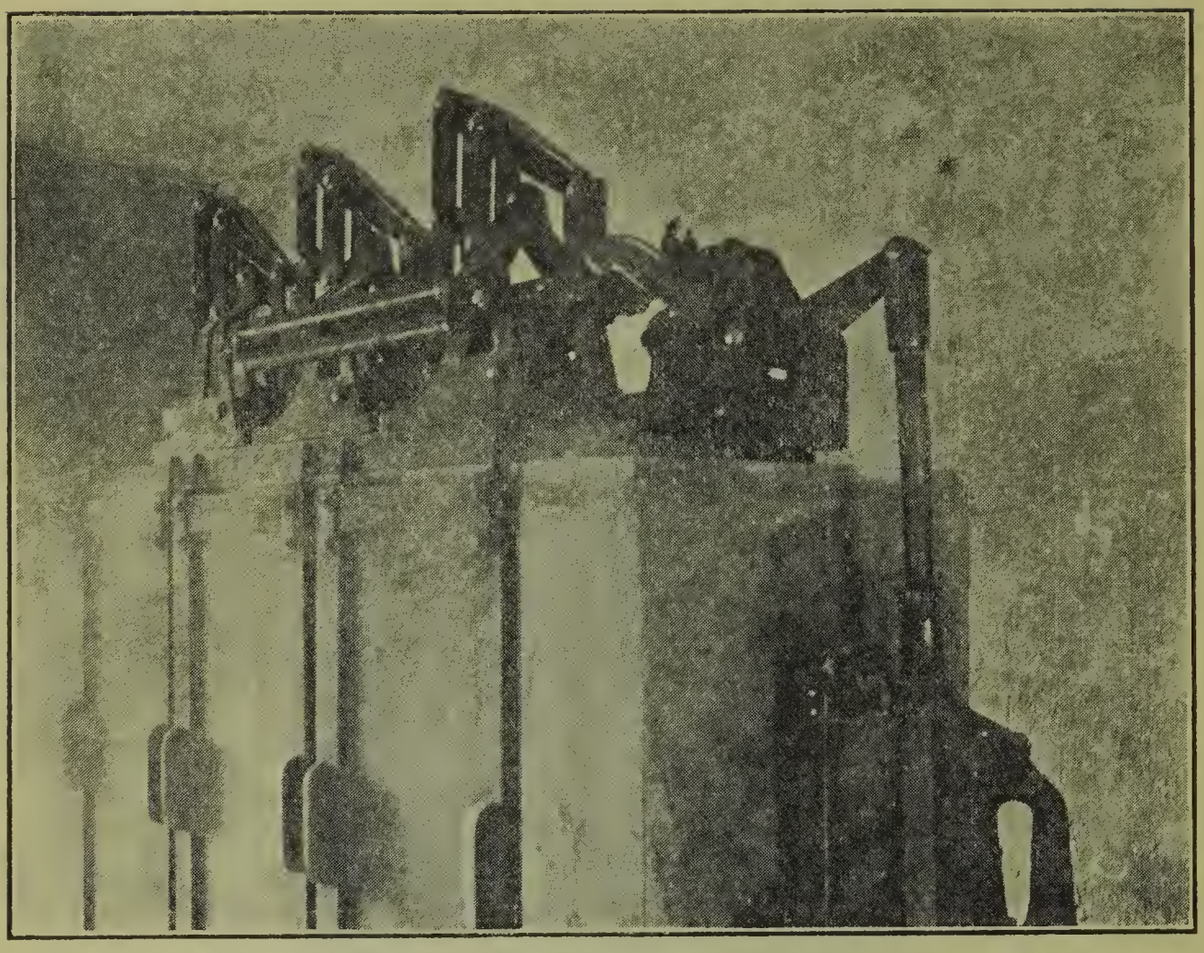

FIG. 19.

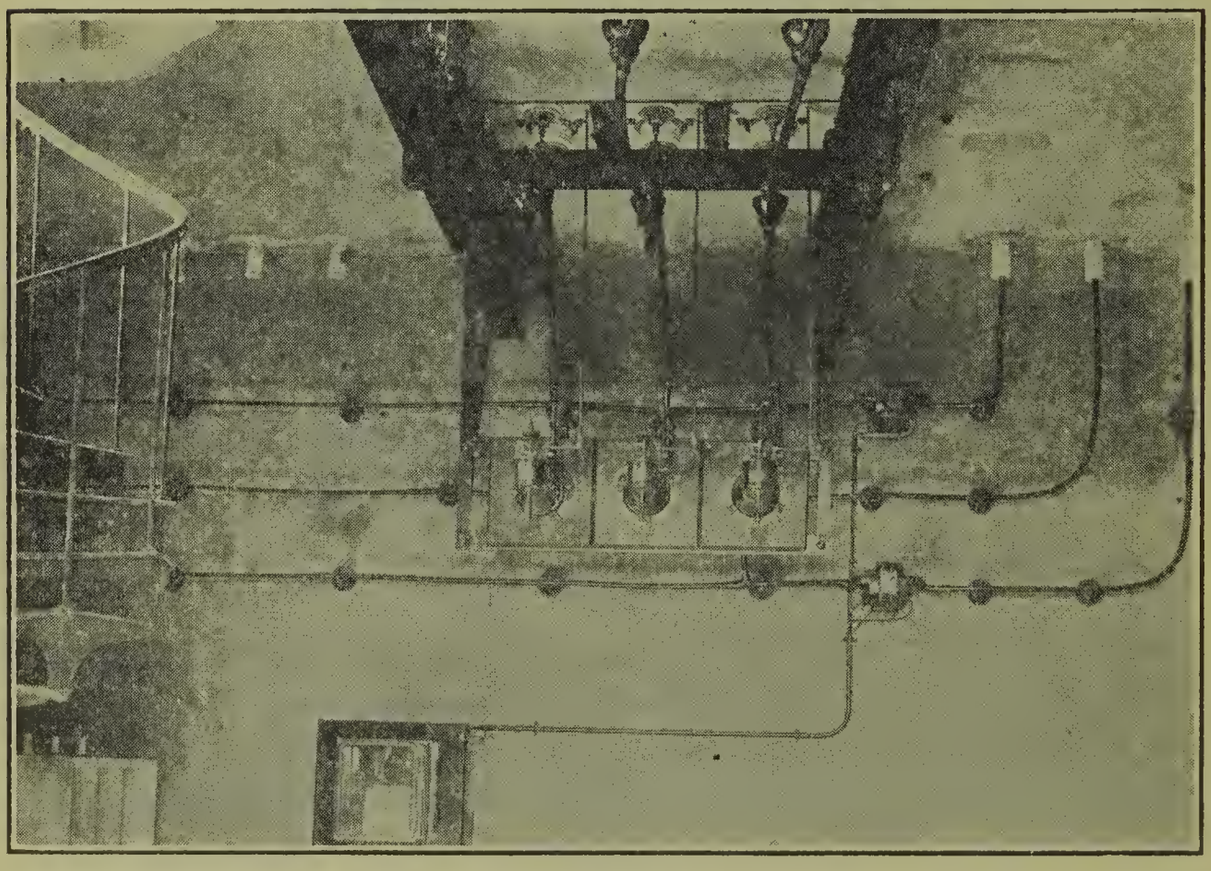

FIG. 20.

On the ground floor and to the right of the knife-switches are located four cells of aluminum lightning-arresters with horn-type spark-gaps, designed and manufactured by the Canadian General Electric Company.

The oil circuit-breaker mentioned above is unique in that the time limit relays for tripping the switches are made to carry the

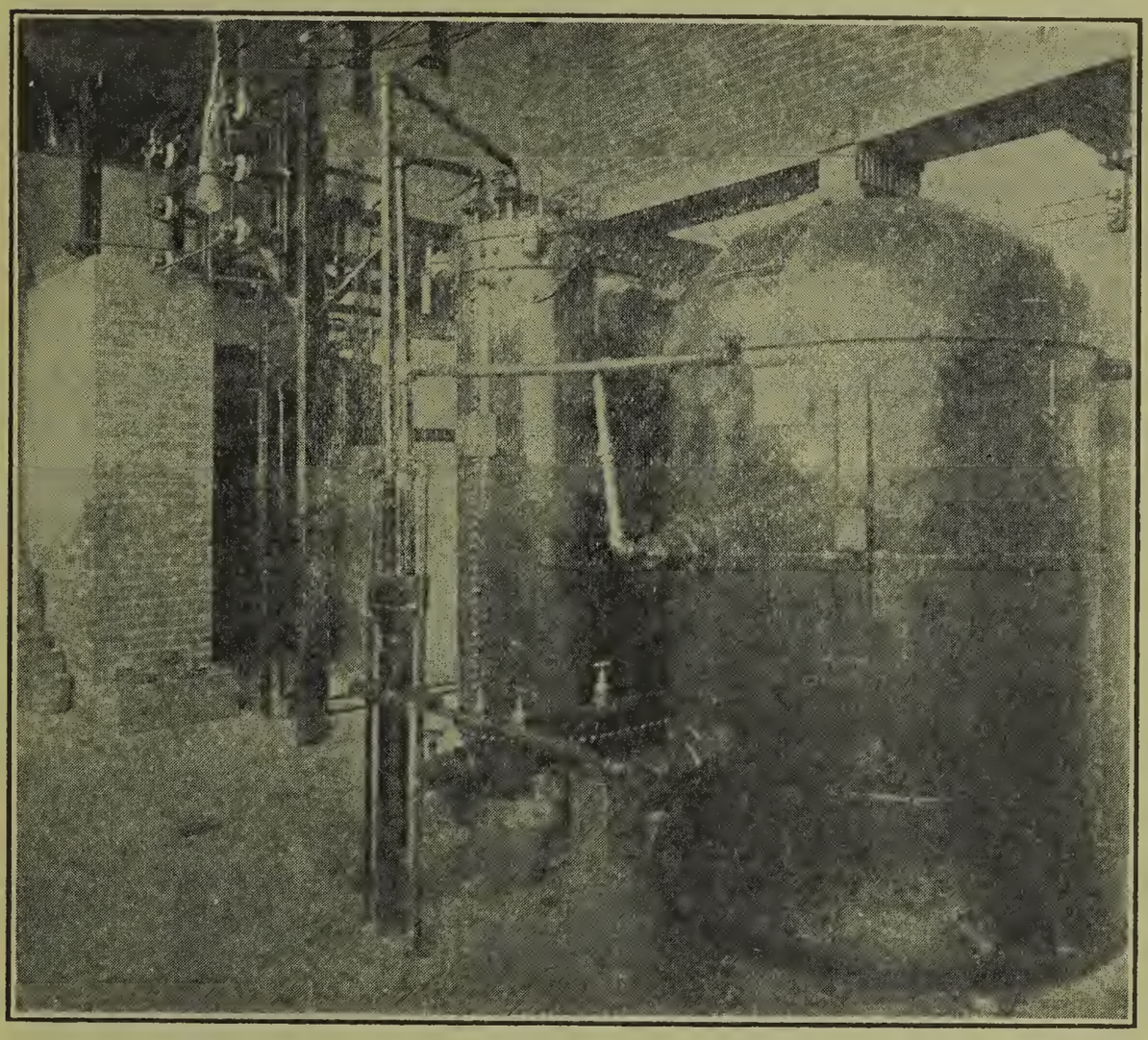

FIG. 21.

full high-voltage current of the line instead of being energized from current transformers (Fig. 20).

The relays are mounted on porcelain insulators directly below the gallery floor, with a tripping-rod passing up through the floor to a toggle-joint on top of the switch. On the floor beneath the gallery in concrete cells are located meter-potential transformers and fuses.

From the oil-switch the circuits pass through a wall to bus-bars in the main transformer room. These bus-bars are constructed

of insulated wire supported on brackets and insulators fastened to the wall adjoining the furnace room (Fig. 21).

The main unit consists of a 750-kw. transformer with induction regulator in secondary circuit. The transformer has a ratio of 12,000 volts to 145 volts, the regulator having a range of 75 volts up or down, which gives a voltage variation on the bus-bars of 75 to 215 volts. Both transformers are of the oil-insulated, water-cooled type, the transformer being built by the Canadian General Electric Company and the regulator by the General Electric Company.

The transformer is protected by a 2-pole, 300-ampere automatic, K4 Canadian General Electric, oil circuit-breaker mounted on an iron framework near the transformer. There are no walls or barriers between the switch-points, which are placed about two feet apart. Knife-switches connect the main unit to the bus-bars, and are arranged so that the transformer can be connected to either bus-bar. The 12,000-volt circuit is opened and closed through a water-switch, whose main elements consist of two wooden barrels supported on porcelain petticoat insulators.

The motor load is provided for by three 40-kw, oil-insulated, self-cooled Packard transformers, with 12,000-volt primaries and 440-volt secondaries connected in *delta*. A 3-pole, 300-ampere, automatic, K4, oil circuit-breaker mounted on pipe framework controls these transformers. A small transformer bearing the name of the Packard Company, with a capacity of 15 kw., 110 to 120-volt secondary, supplies power for lighting the plant. This transformer is protected by out-door Canadian General Electric expulsion fuses. On a small panel in the furnace room are located the indicating meters of the main unit. The switch handles that operate the oil circuit-breakers are mounted on a small marble panel in front of the iron framework carrying the oil pots. The transformer room and main building are of concrete construction. The apparatus was installed by the Norton Company.

This station contains regulating transformers which possess a number of special features. The secondary range of voltage is the greatest, the variation per step the smallest, and the primary voltage the highest of any regulating transformer in the Niagara district. The 11,000-volt transformers found in this station have

been developed by the manufacturers to a point where they may be considered as reliable and satisfactory as the 2,200-volt transformers formerly used for this class of work.

A typical installation of what may be called a converting station may be found in the Hooker Electrochemical Company's installation. This station contains booster type rotaries and concrete constructed switchboard.

Nothing has been said in the paper about the cost of apparatus and efficiencies. I have a few figures here that may be of interest to some of the members; also some points regarding the merits of the different machines for converting alternating current power into direct current power.

Where direct current power is to be obtained from a long distance alternating current system having a varying voltage and somewhat variable frequency, the transformation may be made by either synchronous converters, motor-generator sets with synchronous motors, or motor-generator sets with induction motors.

As regards the relative merits of the different machines it is evident that first place must be given to the synchronous converter as it excels the motor generators in three important points, namely, reliability, cost, and efficiency.

As to reliability, it is simply a case of one machine getting out of order against two in the case of the motor generators. Furthermore, the converters are always wound for a low voltage while the motor generators may be wound for a high voltage and consequently have a lower factor of safety.

The question of cost is also decidedly in favor of the synchronous converter, being again a case of one machine against two. Units of the size found in several of the new Niagara stations, 750 kw., may be installed, complete with switchboards, for about $21.00 per kw., exclusive of buildings, the rotary and transformers costing about $15.00 per killowatt. Units of 100 kw. size will cost about $30.00 per kw. installed. Motor generators of 750 kw. capacity, wound for 11,000 volts, will cost about 15 percent more than synchronous converters of the same size. In large units of 1,500 kw. capacity the cost would be about the

same, as the rotary would probably run at 187 r.p.m. for 25-cycle machines and the motor generator sets, by using an interpole design, at 300 r.p.m.

The overall efficiency of a 750 kw. rotary converter is about 92 percent and a 100 kw. converter 88.5 percent. Motor-generators of the same size will have an overall efficiency 4 or 5 percent lower. If we consider a 750 kw. synchronous converter, operating at 75 percent load and power costing $20.00 per h. p.-year, we find that a 5 percent saving in the power bill is equal to $750 per year which, capitalized at 6 percent, represents $12,500, or in other words, one could pay $16 per kw. more for converters than motor-generators.

Voltage regulation may be accomplished just as easily with synchronous converters as with motor generators. The synchronous converters can be provided with compound windings or automatic field regulators which are responsive to either voltage or current, and it is possible to handle a 10 percent voltage variation on the alternating current side with not more than a ½ percent variation on the direct current side.

Sixteen hundred kw., 25-cycle, regulating transformers with 11,000 volt primary and low voltage secondary may be installed complete, exclusive of buildings, for about $12.00 per kw. Transformers of this class have an efficiency ranging from 95.5 to 98.5 percent, depending on the secondary voltage. Four hundred kw., 25-cycle, regulating transformers cost about $16.00 per kw. installed, with an efficiency of 96.5 to 97.5 percent.

Furnace transformers of 2,000 kw. capacity, 25-cycle, with 11,000-volt primary and 100-volt secondary, are worth $4.00 per kw., installed complete with switchboards and supply cables, the efficiency of such transformers being about 98.5 percent at full load. Small transformers of 300 kw. size will cost close to $7.00 per kw. installed, with an efficiency of 98 percent.

In making an estimate on the cost of apparatus for any electrochemical plant, there are many factors which must be predetermined in order to obtain figures which will exactly apply to any set of local conditions. The figures given above represent the cost per kw. of installations similar to several of those recently completed in the Niagara district.

DISCUSSION.

Mr. F. A. J. FitzGerald: There is one point that is not brought strongly out in Mr. Jones' paper. To the members from Niagara Falls what I am going to say is perfectly well known, but to others I have found by experience that it is not. When people come from places where they have been accustomed to use steam-generated power, and consider the erection of plants at Niagara Falls, they are astonished at some of the apparatus they have to put in. Just as an example of that you might take some plant in Niagara Falls, such as the Norton Company's described on pp. 486 to 490. This plant when it was originally started used 1,000 h. p., but if you turn to the description of the large switch shown in Fig. 19, you find that it has a capacity of 5,000 h. p. What people coming to Niagara Falls fail to realize is the enormous power that is at the back of all their apparatus, and that their apparatus must be such as to protect them from the great currents which are derived from the generators in the power house. Any one keeping this in mind will find this paper very instructive reading and will understand some things that otherwise might appear surprising.

There is one other point. The illustrations show in all cases that a large amount of space is devoted to apparatus. This is a matter in which mistakes are frequently made in designing electric furnaces and purely electrochemical plants. Not enough space is given for the apparatus. This point is most important, and according to our experience it is not even safe to trust the electrical companies to give you a proper design in that regard, because the electrical companies will give you a design with which you are quite satisfied. Then you see Mr. Jones, representing the power company, and he is not at all pleased with the result.

Mr. L. E. Saunders: I think we owe a debt to Mr. Jones for his attempt to complete the record at the present time of the electrical installations in the Niagara district. Those of us who have helped to supply this information know how difficult it is to get such information out of the Niagara manufacturers.

Dr. Edwin F. Northrup: Mr. President, everything at Niagara and at these great power plants is on a gigantic scale,

but I am glad to say that they cannot make currents so large that they are not now easily measured to, perhaps, better than half of one percent. I have just come from the Aluminum Company's works at Massena, and studied for eight hours the operation of one of the ammeters exhibited before this Society. It was measuring 14,000 amperes regularly, with a capacity of 16,500 amperes, without failure in any respect, although the design could have been improved in minor details.

Mr. A. J. Jones (*Communicated*): There has been an increase in the amount of power generated at Niagara Falls since the data for this article was compiled. The figures for September, 1911, are as follows:

Electrochemical processes, 151,000 h. p.; railway service, 62,000 h. p.; lighting, 40,500 h. p.; industrial, 60,500 h. p.; total, 314,000 h p.

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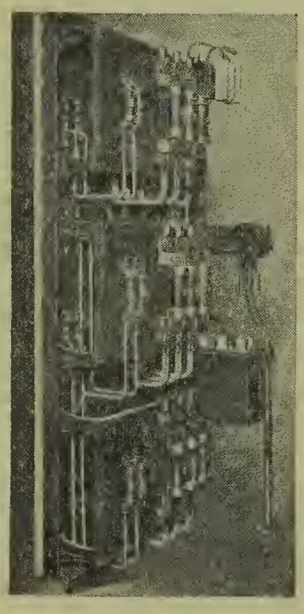

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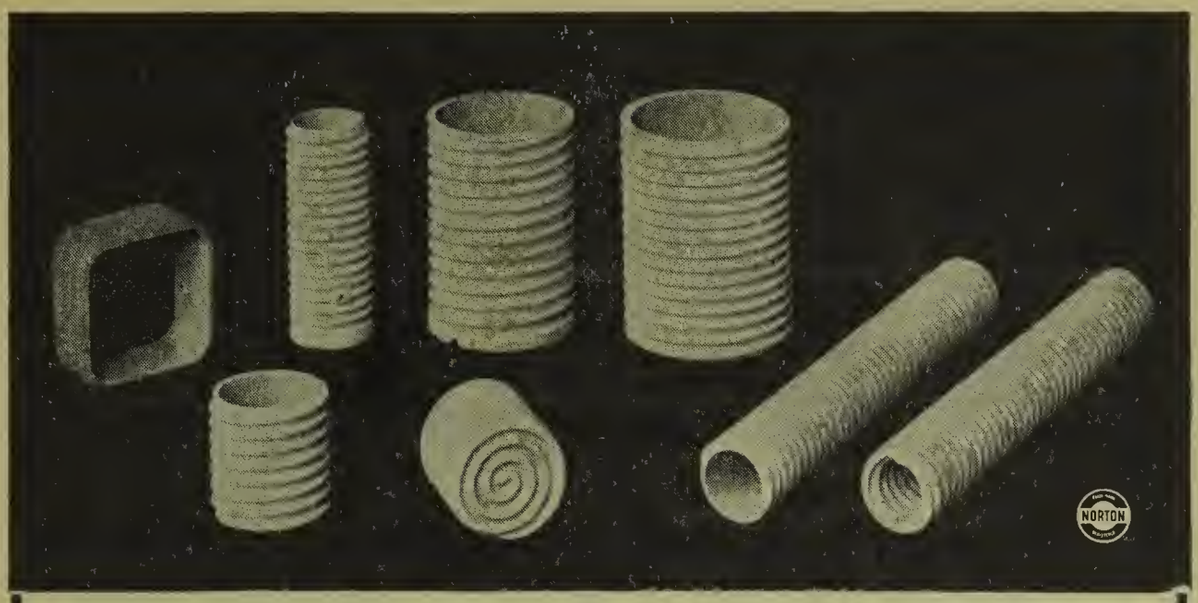

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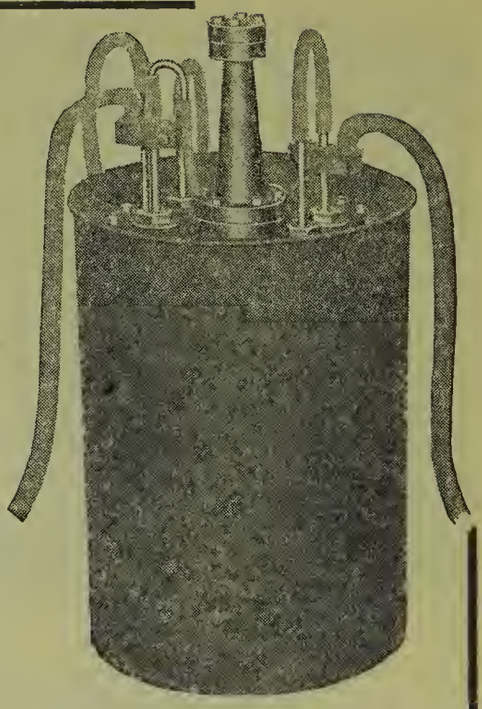

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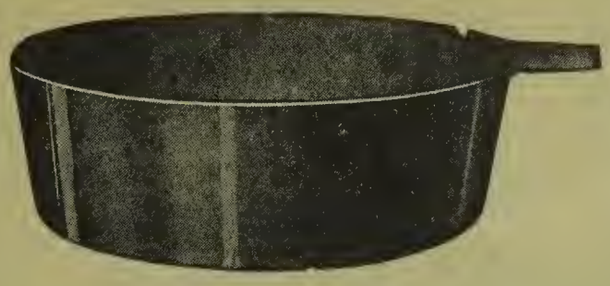

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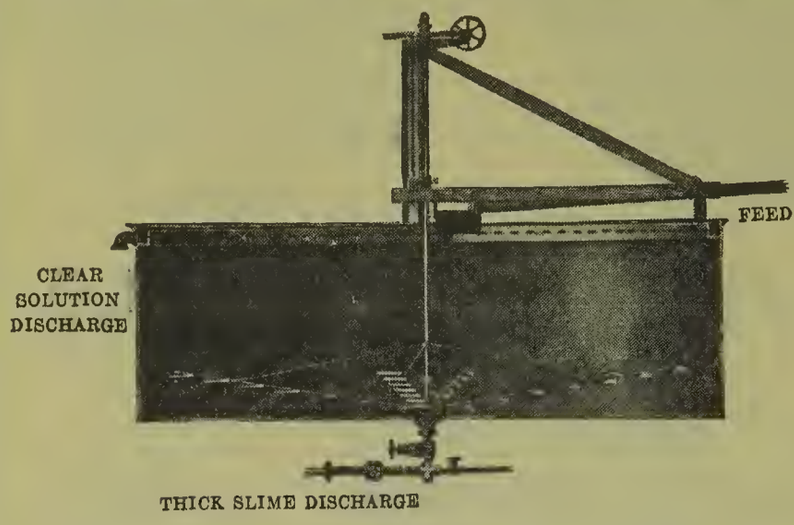

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